



# Assessment and Remediation Of Contaminated Sediments (ARCS) Program

## PILOT-SCALE DEMONSTRATION OF THERMAL DESORPTION FOR THE TREATMENT OF BUFFALO RIVER SEDIMENTS



# **Pilot-Scale Demonstration of Thermal Desorption for the Treatment of Buffalo River Sediments**

**Final Report**

**Prepared by**

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**For the**

**Assessment and Remediation of Contaminated Sediments (ARCS) Program  
U.S. Environmental Protection Agency  
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PILOT-SCALE DEMONSTRATION OF THERMAL DESORPTION FOR THE  
TREATMENT OF BUFFALO RIVER SEDIMENTS

ABSTRACT

This report presents the results of a pilot scale demonstration to remediate contaminated sediments from the Buffalo River. A thermal desorption unit was evaluated for its effectiveness in remediating Buffalo River sediments contaminated with polycyclic aromatic hydrocarbons (PAHs). Sediments were processed at various water contents, thermal unit residence times, and temperatures to evaluate the effect of these process variables on treatment efficiency and materials handling. A portion of the residual solids from the thermal treatment process was mixed with various proportions of Portland cement to evaluate the ability of one solidification/stabilization process to bind metal contaminants.

With sediments remaining in the thermal desorption unit from 30 to 90 minutes and sediment temperatures reaching 300 to 480°F; 43.2 to 97.9 percent of total PAHs were removed while 9.1 to 100 percent of total PCBs (Aroclors 1248 and 1254) were removed. Although this thermal process had little effect on most metals, 16.7 to 100 percent of mercury was removed from sediments during processing. Removal rates for constituents of concern did not correlate well with treatment times or temperatures.

This paper has been reviewed in accordance with the U.S. Environmental Protection Agency's peer and administrative review policies and approved for presentation and publication.

## FINAL REPORT

### PILOT-SCALE DEMONSTRATION OF THERMAL DESORPTION FOR THE TREATMENT OF BUFFALO RIVER SEDIMENTS

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# LIST OF ABBREVIATIONS AND SYMBOLS

## ABBREVIATIONS

amp	ampere
AOC	Area of Concern
ARCS	Assessment and Remediation of Contaminated Sediments
BTU/hr	British Thermal Unit per hour
CDF	Confined Disposal Facility
DSCM	Dry Standard Cubic Meter
EA	Environmental Assessment
ETWG	Engineering Technology Work Group
FONSI	Finding of No Significant Impact
g	Gram
GLNPO	Great Lakes National Program Office
gpm	gallons per minute
HMW	high molecular weight
IJC	International Joint Commission
kg/hr	kilograms per hour
kw	kilowatt
L	liter
lbs	pounds
lb/hr	pounds per hour
lbs/sq in	pounds per square inch
LMW	low molecular weight
min	minute
mg	milligram
ng	nanogram
ng/g	nanogram per gram
NYSDEC	New York State Department of Environmental Conservation
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
ppb	parts per billion
ppm	parts per million
RCRA	Resource Conservation and Recovery Act
RETEC	Remediation Technologies, Inc.
SBLT	sequential batch leach test
scfm	standard cubic feet per minute
TCLP	Toxicity Characteristic Leaching Procedure
TOC	total organic carbon
TSCA	Toxic Substances Control Act
USC	unconfined compressive strength
ug/g	microgram per gram
ug/l	microgram per liter
USEPA	United States Environmental Protection Agency
um	microgram
v	volt
WES	Waterways Experiment Station

## SYMBOLS

Cr	chromium
Cu	copper
°F	degrees Fahrenheit
Hg	mercury
N <sub>2</sub>	nitrogen gas
%	percent
Pb	lead
pH	- log (H <sup>+</sup> )
r	correlation coefficient
Zn	zinc

### **DISCLAIMER**

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# PILOT-SCALE DEMONSTRATION OF THERMAL DESORPTION FOR THE TREATMENT OF BUFFALO RIVER SEDIMENTS

## 1.0 INTRODUCTION

The 1987 amendments to the Clean Water Act, Section 118(c)(3), authorized the United States Environmental Protection Agency's (USEPA) Great Lakes National Program Office (GLNPO) to conduct a 5-year study and demonstration project on the control and removal of toxic pollutants in the Great Lakes, with emphasis on the removal of toxic pollutants from bottom sediments (U.S. Environmental Protection Agency, 1990). The Great Lakes Water Quality Board of the International Joint Commission (IJC) identified 43 Areas of Concern (AOC) in the Great Lakes Basin where one or more of the objectives of the 1978 Great Lakes Water Quality Agreement and other jurisdictional standards, criteria, or guidelines are exceeded. GLNPO initiated the Assessment and Remediation of Contaminated Sediments (ARCS) Program to assess the nature and extent of bottom sediment contamination at the selected AOCs, evaluate and demonstrate remedial options, and provide guidance on the assessment of contaminated sediment problems and the selection and implementation of necessary remedial actions in the AOCs and other locations in the Great Lakes. The Buffalo River AOC, Buffalo, New York, was one area specified in the Clean Water Act as requiring priority consideration in locating and conducting on-site demonstration projects.

Past industrial and municipal discharges to the Buffalo River have polluted the river and its sediments. As a result, the river exhibits environmental degradation and impairment of beneficial uses of water and biota (New York State DEC, 1989). A pilot-scale demonstration was conducted in Buffalo, New York in the fall of 1991 to evaluate the ability of a thermal desorption process to remediate Buffalo River sediments contaminated with polynuclear aromatic hydrocarbons (PAHs).

### 1.1 OBJECTIVE

The objective of the Buffalo River pilot scale treatment technology demonstration was to evaluate thermal desorption as a treatment technology for sediments from the Buffalo River Area of Concern. Specific objectives of the pilot-scale demonstration included determining: the thermal desorption process' efficiencies in removing organic contaminants from sediments; the operating parameters that affect the removal efficiencies; the equipment necessary to achieve those removal efficiencies; the pretreatment handling and processing requirements of the sediments; and the characteristics of each of the process residual streams and the proper method of disposal for each residual. Another objective of the demonstration was to provide technology-specific information to be used in the development of



cost estimates for full scale remediation projects. In addition, a solidification process was evaluated by mixing treated sediments from the thermal desorption process with various proportions of cementitious material. The solidified blocks were sampled, and analyzed to determine the effectiveness of the solidification.

## 1.2 DESCRIPTION OF THE BUFFALO RIVER AREA OF CONCERN

### 1.2.1 Watershed Description

The watershed of Buffalo River and its tributaries, Cayuga, Buffalo, and Cazenovia creeks is located in the west central portion of New York State (Figure 1). The land area is roughly triangular in shape. Buffalo and Cayuga creeks originate in the Allegheny Plateau and flow northwest toward Lake Erie. Buffalo Creek rises near the town of Java and flows northwesterly to its confluence with Cayuga Creek in the town of West Seneca. The drainage area of Buffalo Creek is 150 square miles (New York State DEC, 1989). Cayuga Creek, with a drainage area of 128 square miles, rises near North Java Station and flows westerly through the northern part of the Buffalo River watershed. The confluence of Cayuga Creek and Buffalo Creek form the head of the Buffalo River.

Cazenovia Creek generally flows north from its head waters near Springville, New York to its confluence with the Buffalo River within the Buffalo, New York city limits. The drainage area of Cazenovia Creek is 138 square miles. From Cazenovia Creek, the Buffalo River flows westerly to its mouth at the eastern end of Lake Erie. Overall, the Buffalo River is 8.1 miles in length and its drainage area is approximately 446 square miles.

The Buffalo River and its sediments have been polluted by over 50 years of industrial and municipal discharge and disposal of waste. Fishing and quality of aquatic life within the Area of Concern (Figure 2) have been impaired by heavy metals and polycyclic aromatic hydrocarbons (PAHs) in sediments. Fish and wildlife habitat have been degraded by alterations to the river including modifications to the shoreline such as bulkheading. Levels of metals and cyanides in the sediment prevent open lake disposal of sediments dredged from the river. Other potential sources of pollution to the Buffalo River include inactive hazardous waste sites, combined sewer overflows, and other point and non-point sources of pollution. While the Buffalo River sediments are contaminated, they are not considered "toxic" or "hazardous" based on strict regulatory definitions, and are therefore not subject to the appropriate regulations of the Toxic Substances Control Act (TSCA) or the Resource Conservation and Recovery Act (RCRA).

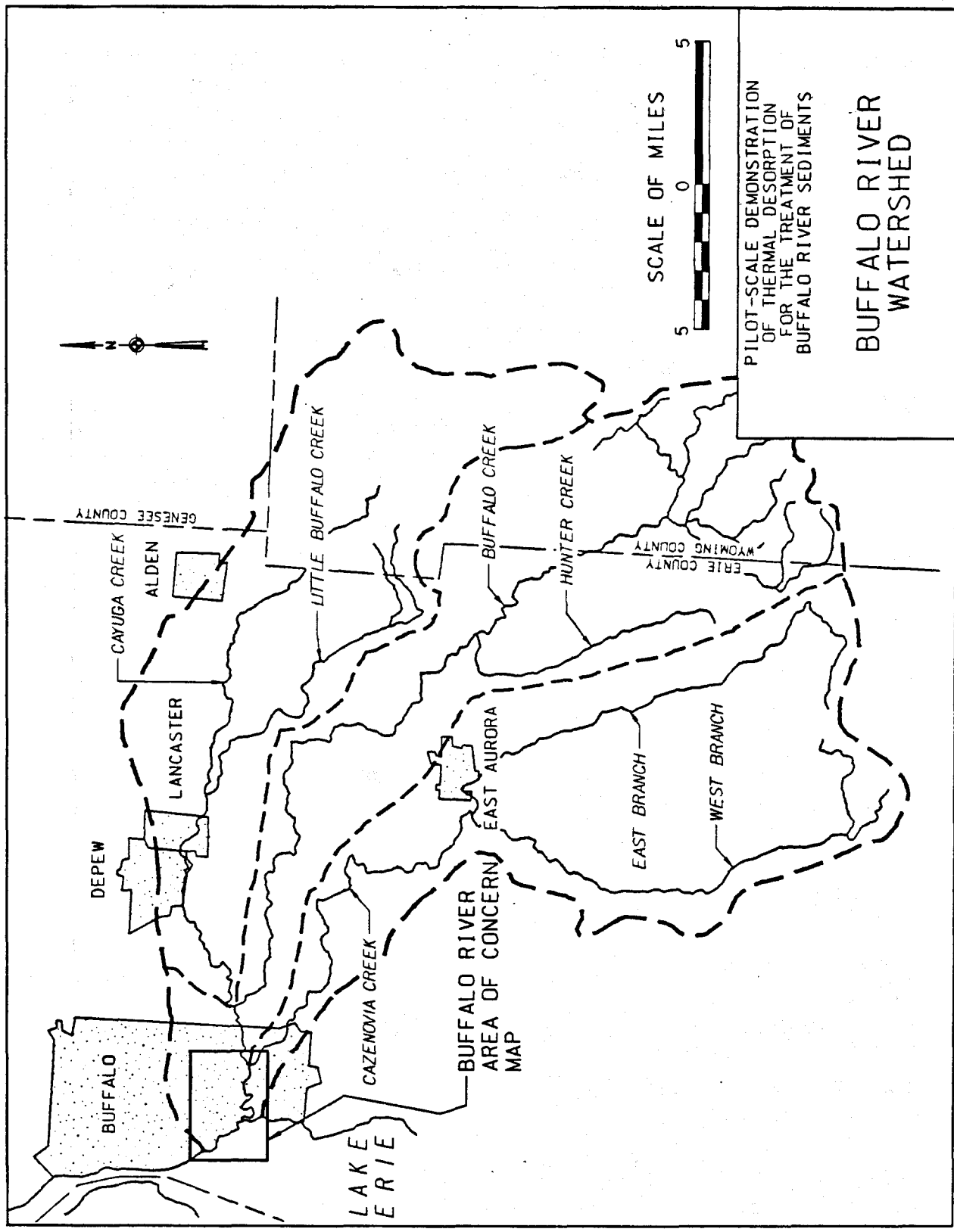


FIGURE 1

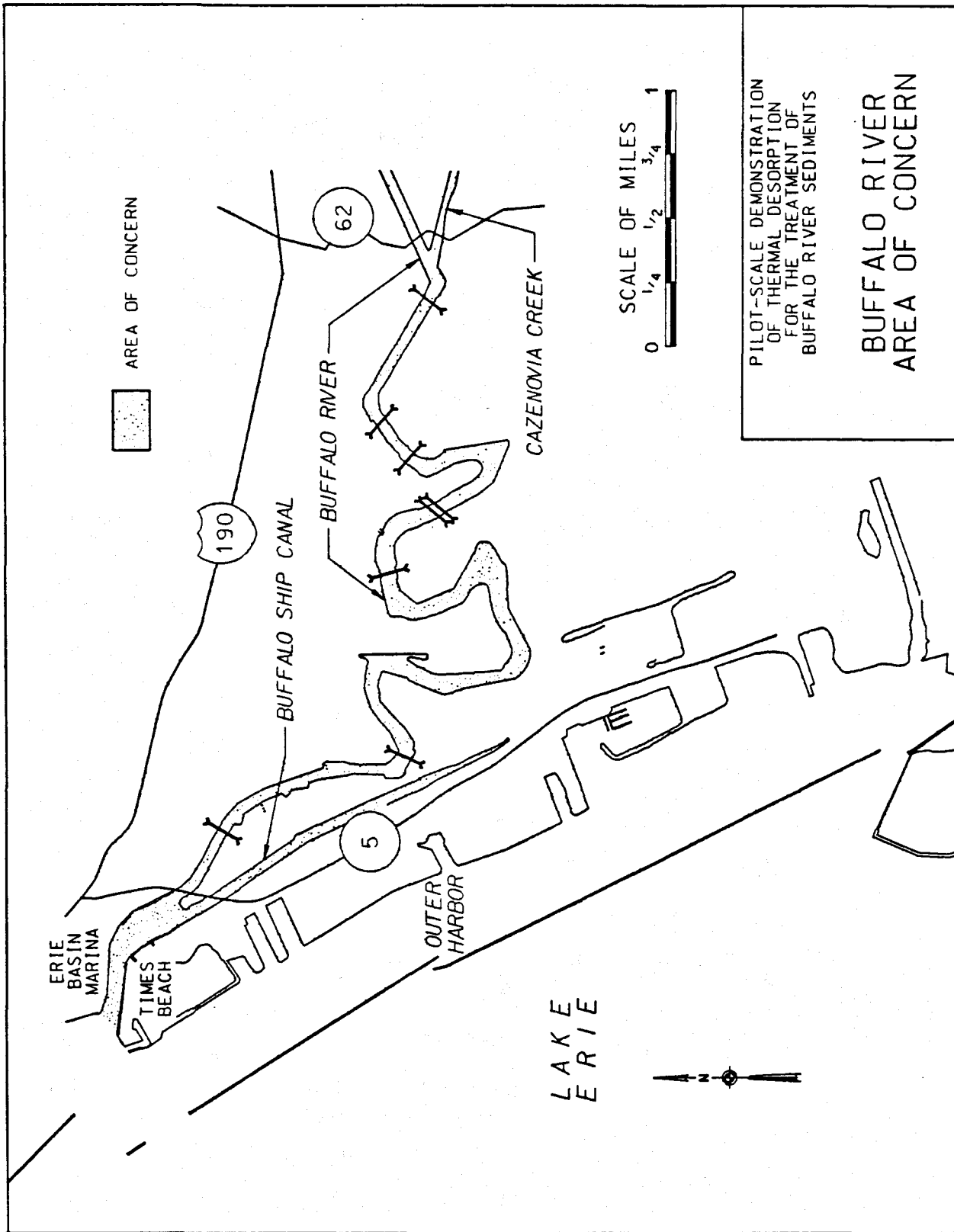


FIGURE 2

### 1.2.2 Status of Remedial Action Plan

New York State Department of Environmental Conservation (NYSDEC) and other Federal, State, and local agencies have and continue to carry out remediation of environmental problems along the Buffalo River. NYSDEC completed and issued the Buffalo River Remedial Action Plan (RAP) in November 1989. The RAP contained initial agency commitments to implement the remedial action strategy. To track implementation of the RAP, NYSDEC has issued annual reports to illustrate the progress on remediation by listing accomplishments of the past year and describing commitments for the current year.

To assist NYSDEC in the remediation process, a Remedial Advisory Committee (RAC) was formed in 1990. The RAC is representative of concerned groups within the community that have an interest in the Buffalo River. These groups include government officials, public interest groups, economic interests, and private citizens.

The following is a brief summary of RAC activities on the Buffalo River. A flow activated sampling station was established by NYSDEC to assist in stream water quality monitoring (New York State Department of Environmental Conservation, 1992). Event related sampling has been undertaken and will be continued into 1993. Sediment transport modeling is being conducted by the USEPA under the ARCS program. A dredging demonstration was conducted in 1992 by the Corps of Engineers to evaluate the efficiencies of several dredge types. Phase I investigations for all 36 inactive hazardous waste sites have been completed, while all but seven Phase II investigations have been completed. Remedial Investigation/Feasibility Studies (RI/FS) were completed for three sites in 1991-92, while two additional RI/FS's are underway. A combined sewer system model has been developed and verified for the main interceptors of the Buffalo Sewer Authority collection system. Operational simulations have been undertaken and cost estimates of alternatives for overflow reduction/treatment have been developed. A plan to assess fish and wildlife habitat conditions and improvement potential has been developed. Habitat assessment field work has been initiated by NYSDEC and will be completed in 1993.

### 1.2.3 Sediment Physical/Chemical Character

#### 1.2.3.1 Sources of Sediments--

The major source of sediment in the Buffalo River is in runoff from the surrounding watershed. Depending on factors such as river velocities and discharge, channel topography, bank erosion and wind, much of the sediment originating in runoff is either deposited in the river channel bottom or is carried to areas further downstream. A large portion of this sediment accumulates in the Buffalo River Federal Navigation Channel.

#### 1.2.3.2 Sediment Pollution--

The Buffalo River watershed is comprised of three major streams which converge at or along its mainstem: Cayuga, Buffalo, and Cazenovia creeks. Within the watershed major land usage is industrial and commercial, with some agricultural usage. Flows into the Buffalo River watershed originate in part from a variety of point and non-point source industrial activities in the watershed, including inactive hazardous waste sites and combined sewer outflows/municipal waste discharges (New York State D.E.C., 1989). These sources contribute to the bottom sediment contamination in the river. Polynuclear aromatic hydrocarbons and metals are contaminants of particular concern in Buffalo River sediments.

#### 1.2.3.3 Sediment Characteristics and Quality--

Historic and recent sediment particle size analyses indicate that bottom sediments within the Buffalo River are comprised of silts and clays, with some sands. Particle size and chemical (inorganic and organic) analyses and 96-hour acute toxicity tests (bioassays) were performed on surface grab samples obtained from the Buffalo River Federal Navigation Channel in 1989 (Aqua Tech Environmental Consultants, 1989). Particle size analysis of the sediment samples indicates they consist primarily of silts and clays (approximately 65 to 99 percent), with some sands (approximately 1 to 35 percent). Regarding inorganic sediment contamination, the results of bulk inorganic analysis performed under the 1989 program showed that most of the sediments were contaminated with elevated levels of numerous metals, including arsenic, barium, copper, iron, manganese, nickel, and zinc (Table 1). The 1989 sediment testing program included analyses for volatile organics, PAHs and polychlorinated biphenyls (PCBs). Table 2 summarizes volatile organics data on sediments. Generally, volatile organics were not detected in sediments with the exception of low levels of 1,3-Dichlorobenzene and high levels of toluene on portions of the Buffalo River. PAH levels, shown in Table 3, ranged from non-detectable to about 2.4 micrograms per gram (ug/g) (benzo(b)fluoranthene). Total PAHs ranged from 5.44 to 12.15 ug/g. PCB and pesticide data summarized in Table 4 show non-detectable levels in the sediments.

The USEPA's Large Lakes Research Station of Grosse Ile, Michigan sampled sediments along the Buffalo River and Buffalo Ship Canal in 1989, 1990, and 1991 with a 4-inch diameter vibracore unit. Results from testing performed on samples collected outside the navigation channel in 1989 show concentration levels for 12 metals at 10 sites along the Buffalo River (Figure 3 and Table 5). Concentration levels for chromium (Cr) ranged from less than 13 ug/g to 312 ug/g while concentration levels for mercury (Hg) ranged from 0.0109 to 1.93 ug/g. Lead (Pb) concentrations ranged from 28 to 314 ug/g while zinc (Zn) concentrations ranged from 32 to 900 ug/g. In general, the highest concentration levels of metals were in the terminal end of the Buffalo Ship Canal and in the middle third reach of the Buffalo River. Test results

TABLE 1  
INORGANIC ANALYSIS OF SURFACE SEDIMENT GRAB SAMPLES  
ug/g (dry)

Bulk inorganic analysis of surface sediment grab samples collected from Buffalo River, Erie County, New York. Sediment sampling areas are shown in Figure 4.

Inorganic Parameter	Sediment Sampling Areas				
	Deadman's Creek	Hamburg Street	Blue Tower Turning Basin	Mobil Oil	
ARSENIC, TOTAL, AS	13	11	10	9	10
BARIUM, TOTAL, BA	90	84	79	91	78
CADMIUM, TOTAL, CD	0.5	1	1	0.6	1
CHROMIUM, TOTAL, CR	18	13	13	9	2
COPPER, TOTAL, CU	49	46	48	40	35
IRON, TOTAL, FE	30300	29200	28400	30800	24300
LEAD, TOTAL PB	82	66	62	55	60
MANGANESE, TOTAL, MN	490	490	460	480	520
MERCURY, TOTAL, HG	0.40	0.37	0.34	0.28	0.24
NICKEL, TOTAL, NI	31	30	29	32	25
RESIDUE, TOTAL (TS), %	48.7	53.4	55.2	50.6	57.0
SELENIUM, TOTAL, SE	<1	<2	<2	<2	<2
SILVER, TOTAL, AG	<0.5	<0.5	<0.6	<0.6	<0.6
SODIUM, TOTAL, NA	440	450	440	500	430
SPECIFIC GRAVITY	1.41	1.5	1.48	1.48	1.56
CARBON, TOTAL ORGANIC, C	1400	1200	1000	1200	1100
ZINC, TOTAL, ZN	210	210	180	170	120

DATA SOURCE: Referenced Aqua Tech Report

TABLE 2  
VOLATILE ORGANICS DATA FOR SURFACE SEDIMENT GRAB SAMPLES  
ug/g (dry)  
Volatile Organics data on surface sediment grab samples  
collected from Buffalo River, Erie County, New York.  
Sediment sampling areas are shown in Figure 4.

Volatile Organic	Sediment Sampling Areas					
	Deadman's Creek	Hamburg Street	Blue Tower Turning Basin	Mobil Oil		
Acrolein	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Acrylonitrile	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Benzene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Bromoform	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
Carbon Tetrachloride	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Chlorobenzene	0.040	< 0.005	0.019	< 0.005	0.034	< 0.005
Chlorodibromomethane	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
Chloroethane	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
2-Chloroethyl Vinyl Ether	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
Chloroform	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Dichlorobromomethane	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Dichlorodifluoromethane	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
1,1-Dichloroethane	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
1,2-Dichloroethane	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
1,1-Dichloroethene	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
1,2-Dichloropropane	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
cis-1,3-Dichloropropene	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
trans-1,2-Dichloropropene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Ethyl Benzene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Methyl Bromide	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Methyl Chloride	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Methylene Chloride	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
1,1,2,2-Tetrachloroethane	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
Tetrachloroethene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Toluene	< 0.010	< 0.010	< 0.010	< 0.010	9.77	12.9
cis-1,2-Dichloroethene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
trans-1,2-Dichloroethene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
1,1,1-Trichloroethane	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
1,1,2-Trichloroethane	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Trichloroethene	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Trichlorofluoromethane	< 0.020	< 0.020	< 0.020	< 0.020	< 0.020	< 0.020
Vinyl Chloride	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050	< 0.050
Total Xylenes	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010
1,3-Dichlorobenzene	0.249	0.374	0.048	0.383	0.263	< 0.005
Other Volatile Hydrocarbons *	3.52	2.65	1.41	1.83	4.09	4.28

\* Concentration estimate based on response of internal standard.

DATA SOURCE: Referenced Aqua Tech Report

TABLE 3  
PAH DATA FOR SURFACE SEDIMENT GRAB SAMPLES  
ug/g (dry)

PAH data on surface sediment grab samples collected  
from Buffalo River, Erie County, New York. Sediment  
sampling areas are shown in Figure 4.

PAH	Sediment Sampling Areas					
	Deadman's Creek	Hamburg Street	Blue Tower Turning Basin	Mobil Oil		
Acenaphthene	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
Acenaphthylene	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
Anthracene	0.13	0.34	0.16	0.13	0.14	0.12
Benzo(a)Anthracene	0.58	0.93	0.51	0.45	0.46	0.39
Benzo(a)Pyrene	0.79	1.37	0.81	0.86	0.74	0.68
Benzo(b)Fluoranthene	1.58	2.38	1.45	1.49	1.21	1.11
Benzo(ghi)Perylene	<0.40	0.49	<0.40	<0.40	<0.40	<0.40
Benzo(k)Fluoranthene	<0.20	<0.20	<0.20	<0.20	<0.20	<0.20
Chrysene	0.73	0.98	0.57	0.63	0.52	0.43
Dibenzo(a,h)Anthracene	<0.40	<0.40	<0.40	<0.40	<0.40	<0.40
Fluoranthene	1.36	2.33	1.30	1.21	1.05	1.21
Fluorene	<0.30	<0.30	<0.30	<0.30	<0.30	<0.30
Indeno(1,2,3-cd)Pyrene	0.44	0.61	0.41	<0.30	<0.30	<0.30
Naphthalene	<0.30	<0.30	<0.30	<0.30	<0.30	<0.30
Phenanthrene	0.58	1.34	0.59	0.59	0.60	0.78
Pyrene	0.83	1.38	0.72	0.76	0.83	0.72
Total PAHs	7.02	12.15	6.52	6.12	5.55	5.44

DATA SOURCE: Referenced Aqua Tech Report



TABLE 4  
 PESTICIDE AND PCB DATA FOR SURFACE SEDIMENT GRAB SAMPLES  
 ug/g (dry)  
 Pesticide and PCB data on surface sediment grab samples  
 collected from Buffalo River, Erie County, New York.  
 Sediment sampling areas are shown in Figure 4.

Pesticides and PCB	Sediment Sampling Areas				
	Deadman's Creek	Hamburg Street	Blue Tower Turning Basin	Mobil Oil	
Aldrin	<0.02	<0.02	<0.02	<0.02	<0.02
alpha-BHC	<0.02	<0.02	<0.02	<0.02	<0.02
beta-BHC	<0.02	<0.02	<0.02	<0.02	<0.02
gamma-BHC	<0.02	<0.02	<0.02	<0.02	<0.02
delta-BHC	<0.02	<0.02	<0.02	<0.02	<0.02
Chlordane	<0.10	<0.10	<0.10	<0.10	<0.10
4,4'-DDD	<0.02	<0.02	<0.02	<0.02	<0.02
4,4'-DDE	<0.03	<0.03	<0.03	<0.03	<0.03
4,4'-DDT	<0.03	<0.03	<0.03	<0.03	<0.03
Dieldrin	<0.02	<0.02	<0.02	<0.02	<0.02
Endosulfan I	<0.03	<0.03	<0.03	<0.03	<0.03
Endosulfan II	<0.03	<0.03	<0.03	<0.03	<0.03
Endosulfan Sulfate	<0.03	<0.03	<0.03	<0.03	<0.03
Endrin	<0.05	<0.05	<0.05	<0.05	<0.05
Endrin Aldehyde	<0.05	<0.05	0.06	<0.05	<0.05
Heptachlor	<0.03	<0.03	<0.03	<0.03	<0.03
Heptachlor Epoxide	<0.03	<0.03	<0.03	<0.03	0.05
Toxaphene	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1016	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1221	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1232	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1242	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1248	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1254	<0.10	<0.10	<0.10	<0.10	<0.10
PCB-1260	<0.10	<0.10	<0.10	<0.10	<0.10

DATA SOURCE: Referenced Aqua Tech Report

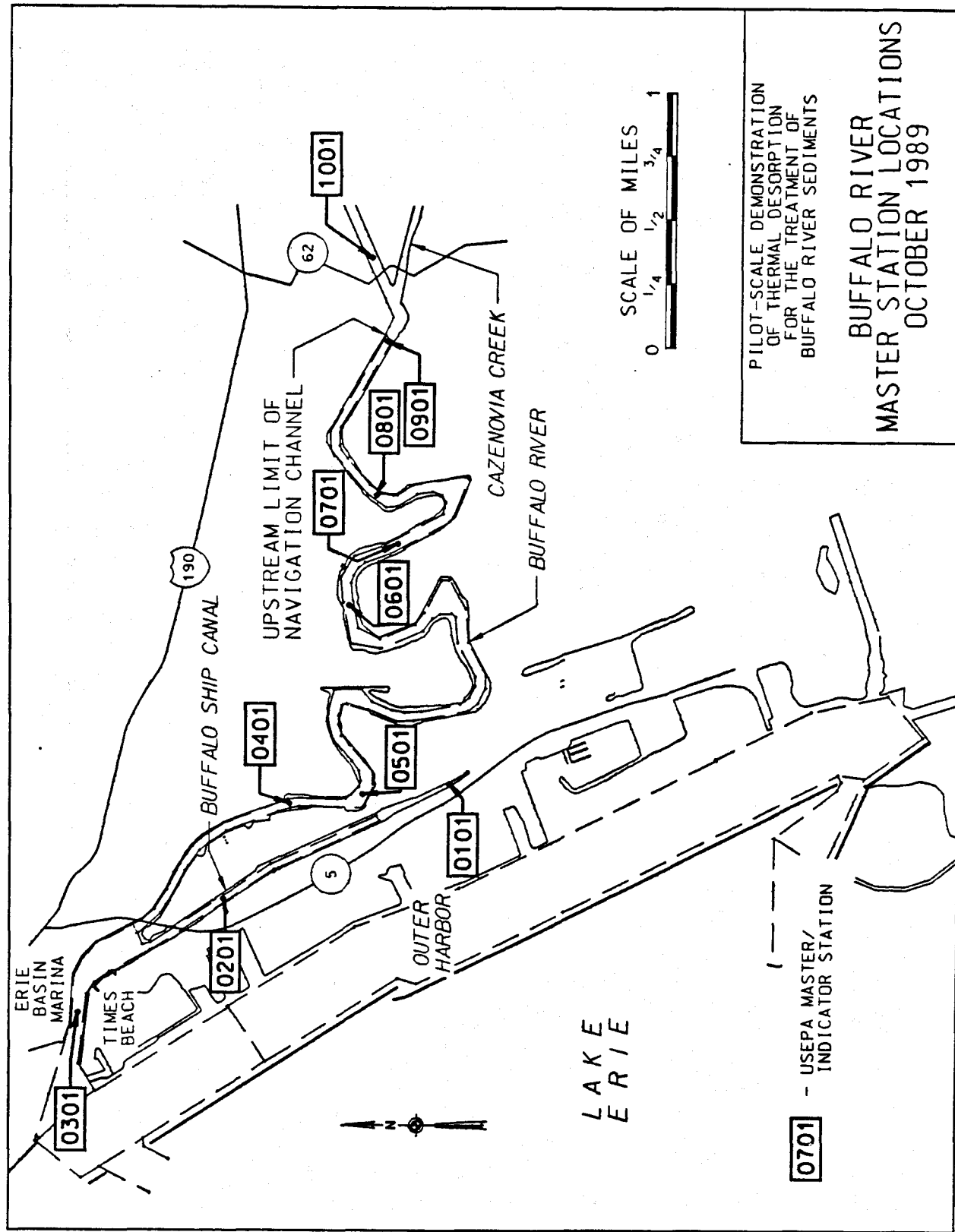


FIGURE 3

TABLE 5

CONCENTRATIONS OF METALS IN TEN BUFFALO RIVER  
SEDIMENT SAMPLES (OCTOBER, 1989)  
ug/g (dry) (except % Fe)  
SAMPLING LOCATIONS ARE SHOWN IN FIGURE 3

Sample	Ag	As	Cd	Cr	Cu	%Fe	Hg	Mn	Ni	Pb	Se	Zn
0101	0.46	34	4.0	312	148	5.5	1.93	1,386	57	286	3.8	900
0201	<0.03	<1.4	0.035	<13	8.2	0.33	0.019	40	5.2	28	<0.49	32
0301	0.44	13	1.4	113	67	4.4	0.624	685	45	107	<0.92	286
0401	0.22	12	1.0	77	50	4.2	0.186	790	50	67	<0.85	220
0501	0.16	<4.5	1.6	100	60	5.4	0.329	673	47	314	<1.0	371
0601, Rep 1 (a)	0.21	13	1.2	110	90	4.2	1.62	630	52	140	0.93	390
0601, Rep 2 (a)	0.24	12	1.2	130	93	4.2	1.76	630	46	150	<0.89	390
0701	0.13	12	0.90	92	49	4.1	0.233	730	44	70	<0.86	200
0801	0.13	12	0.70	70	46	3.7	0.132	730	43	51	<0.88	170
0901	0.12	11	0.69	56	41	3.4	0.066	730	40	49	<0.84	160
1001	0.12	8.2	0.57	46	35	3.0	0.082	560	34	43	<0.83	140

(a) Duplicate Samples Collected at Buffalo River Station 0601  
DATA SOURCE: Environmental Protection Agency (Unpublished)

for 20 organic parameters (PAHs) analyzed at 9 of the 10 sampling sites are given in Table 6. Generally the highest concentration of PAHs were at sample site 0601 in the Buffalo River and 0101 at the terminal end of the Buffalo Ship Canal. Benzo(a)pyrene concentrations ranged from undetectable at 54 nanogram per gram (ng/g) to 2500 (ng/g) at sample site 0601.

In 1990 and 1991 sediment vibracore samples were collected by USEPA in the Buffalo River. Generally, approximately 1 to 3 meter core samples were taken and analyzed. Analytical results indicated that sediment contamination is either (1) relatively low and consistent with respect to depth, or (2) increases with respect to depth to a maximum level at which point a relatively clean, natural lacustrine clay layer is reached (U.S. Army Engineer District, Buffalo, 1992).

Areas sampled during the 1991 program are shown on Figure 4. Results of the 1991 sampling revealed that, in general, lightly to moderately polluted sediments overlay heavily polluted sediments as shown in concentrations of chromium, lead, zinc, and PAHs. Some of the core samples extended through the heavily polluted sediments into underlying moderately and lightly polluted sediments at core depths of roughly 3 to 4 meters. Many of the vibracore samples met refusal at a depth of 3 meters or less and did not appear to penetrate deep enough to extend through the heavily polluted sediments and into the underlying moderately and lightly polluted sediments.

## 2.0 DEMONSTRATION APPROACH

### 2.1 TECHNOLOGY SELECTION

A literature review of treatment technologies was performed for the ARCS Program by the Corps of Engineers Waterways Experiment Station (WES) and was used to screen process options for biological, chemical, extraction, immobilization, radiant energy, and thermal technologies (Averett, 1990b). Each process option was assessed on the basis of effectiveness, implementability, and cost. A number of the higher cost thermal processes were eliminated from consideration due to the expense of these processes while numerous other processes were eliminated from further consideration because of the lack of research and development for application to a specific sediment and associated contaminant matrix. The availability of a mobile pilot scale unit was essential for implementing an on-site pilot demonstration. Based on these criteria, a list of those processes that should be retained for demonstration consideration was developed.

A matrix was developed for the processes recommended for consideration for the pilot scale demonstrations, the principal contaminants treatable by each process, and the Areas of Concern

TABLE 6

CONCENTRATIONS OF POLYCYCLIC AROMATIC HYDROCARBONS  
IN TEN BUFFALO RIVER SEDIMENT SAMPLES (ng/g)  
SAMPLING LOCATIONS ARE SHOWN IN FIGURE 3

Compound	0101	0201	0301	0401	0501	0601	0701	0801	0901	1001
<u>Polycyclic Aromatic Hydrocarbons:</u>										
Naphthalene	8600	U57(a)	360	U35	140	830	150	U38	U30	(b)
2-methylnaphthalene	1000	U57	180	U35	150	2200	U42	U38	U30	
Acenaphthylene	670	U61	U50	U37	U45	U45	U45	U40	U31	
Acenaphthene	300	U61	U50	U37	U45	750	U45	U40	U31	
Fluorene	770	U61	310	U37	300	3600	160	U40	U31	
Phenanthrene	2600	U71	1100	300	2100	11000	750	370	380	
Anthracene	720	U68	810	88	500	4500	260	96	U35	
Fluoranthene	3200	U110	1400	620	2100	5400	1100	610	840	
Pyrene	2600	U140	1600	460	1900	7100	1200	560	530	
Benzo(a)anthracene	1500	U43	520	240	690	1900	520	210	220	
Chrysene	1700	U54	810	320	980	2700	770	360	330	
Benzo(b)fluoranthene	3000	U61	770	330	1100	1600	740	440	430	
Benzo(k)fluoranthene	4000	U82	690	340	970	1600	660	300	320	
Benzo(a)pyrene	2500	U54	360	320	930	1300	690	280	310	
Indeno(1,2,3-c,d)pyrene	1600	U89	400	U54	500	1000	270	U60	160	
Dibenzo(a,h)anthracene	350	U68	U56	U41	U50	U50	U50	U45	U35	
Benzo(g,h,i)perylene	1600	U110	470	U67	370	1100	290	U74	170	

(a) - U57 Indicates a Sample Concentration Below the Detection Limit of 57 PPB

(b) - Data Not Yet Available

DATA SOURCE: Environmental Protection Agency (Unpublished)

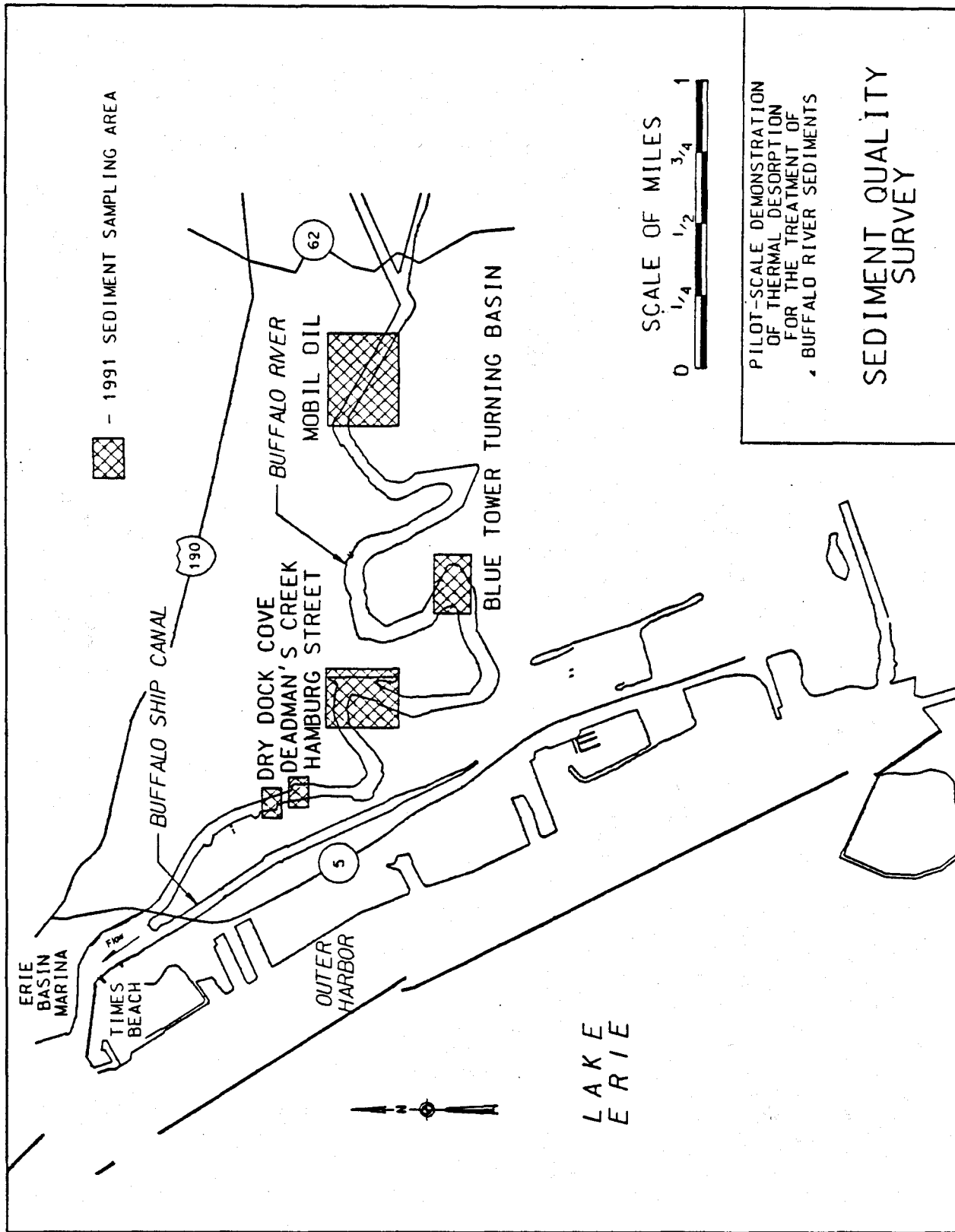


FIGURE 4

where such contaminants are present and the processes are applicable (Averett, 1990a). A list of potential pilot projects was then prepared and these alternatives were ranked for consideration based on factors affecting their selection.

All five priority sites, Ashtabula River, Ohio; Buffalo River, New York; Grand Calumet River, Indiana; Saginaw River, Michigan; and Sheboygan Harbor, Wisconsin are contaminated by organic compounds. Most of these sites have areas of elevated contamination that could be used for a demonstration project. Rather than strictly following the numeric ranking of the potential pilot scale demonstrations, the ARCS Engineering/Technology Work Group (ETWG), responsible for recommending and implementing the demonstration, determined that a variety of the technology groups (biological, chemical, extraction, immobilization, thermal) should be selected for demonstration. With this in mind, thermal desorption, a technology suitable for treating materials contaminated with organics, was selected for a pilot scale demonstration to be conducted at the Buffalo River Area of Concern.

## 2.2 PLANNING DOCUMENT

Buffalo District initiated its planning for the pilot-scale demonstration in the fall of 1990. Coordination efforts associated with the demonstration involved meetings, correspondence, and telephone conversations with representatives of USEPA Region II, NYSDEC, members of the Buffalo River Remedial Action Committee, as well as the Corps of Engineers Regulatory personnel. A meeting was held with NYSDEC personnel in September 1990, while the October 1990 meeting of the ARCS ETWG was held at the NYSDEC Region 9 headquarters in Buffalo, New York. Topics discussed in these meetings included the thermal desorption technology, the scope and proposed location of the demonstration, anticipated treatment process residuals and their disposal and regulatory requirements. A document entitled "Work Plan for Pilot-Scale Demonstration for Remediation of Contaminated Sediments at the Buffalo River Area of Concern," was completed in February 1991. This report addressed sediment quality, description of the Buffalo River AOC, selection of the treatment technology, description of the demonstration, ranging from sediment removal and transport to residuals management and an estimate of all costs associated with the project. Also included was a description of activities associated with the demonstration ranging from regulatory and contractual requirements to monitoring requirements and report preparation. Members of the ETWG, including USEPA Region II, State and local representatives, were given the opportunity to review and comment on the document. Comments were reflected in the final document which was approved by the ETWG.

## 2.3 ENVIRONMENTAL ASSESSMENT

In early 1991, the Corps of Engineers Buffalo District initiated work on an Environmental Assessment (EA). The assessment provided background information and addressed the environmental impacts and statutory compliance of the project. Social impacts were discussed as were the effects of the project on natural resources including air and water quality, aquatic and terrestrial habitat, and threatened and endangered species. A brief description of the various environmental regulations and statutes applicable to the demonstration project and the degree to which the project was in compliance with those statutes and regulations was provided. Since no new dredged materials were to be discharged below Low Water Datum and given the small scope of the project, it was determined that the dredging of the sediments to be remediated fell within the limits specified under Section 10 of the Clean Water Act (Nationwide Permit No. 19) and complied with the provisions of Section 404(b) of the Clean Water Act. A 404(b)(1) Evaluation was not necessary since the discharge of dredged material from the Buffalo Harbor into confined disposal facility No. 4 is covered under the 404(b)(1) Evaluation and Water Quality Certificate prepared for the construction of the disposal facility and any subsequent dredging connected with the facility. The project was also found to be in compliance with the River and Harbor Act of 1970, the Endangered Species Act of 1973, as amended, and numerous other applicable acts. With the dissemination and distribution of the EA and its associated Finding of No Significant Impact (FONSI) to numerous Federal, State, and local agencies and individuals, the project was found to be in full compliance with the National Environmental Policy Act. The FONSI was completed and signed on August 19, 1991.

## 2.4 SCOPE OF WORK/CONTRACT

A scope of work was prepared for a pilot scale demonstration of the remediation of contaminated sediments at the Buffalo River Area of Concern utilizing thermal desorption. This scope of work provided background information, stated the objective of the demonstration, and provided a detailed description of the services required. The scope of work was made a part of the request for proposals that was provided to 36 interested firms responding to an announcement published in the Commerce Business Daily. The two proposals submitted in response to the request for proposals were reviewed by members of the ETWG to determine the prospective firms abilities to conduct the required services. The technical proposals were evaluated using a rating system based on the technical evaluation criteria developed by the ETWG and presented in the Commerce Business Daily announcement. Contract award was based on a firm's ability to meet the technical requirements of the testing involved, the company's qualifications and experience in conducting similar studies, the uniqueness and innovativeness of the technology in treating Great Lakes sediments, a comparison of cost estimates,



and the feasibility of conducting a full scale remediation project with the contractor's technology. After this evaluation was performed, a contract was awarded to Remediation Technologies, Inc. (RETEC) of Concord, Massachusetts.

## 2.5 SAMPLE LOCATION AND EXCAVATION

Based on available sediment sampling and analysis data obtained by the USEPA from 1989 and 1990 sampling operations, there were two areas of the Buffalo River Area of Concern where the sediments generally had higher concentrations of PAHs, than remaining portions of the river. These two areas were near the terminal end of the Buffalo Ship Canal and in an area of the upper portion of the Buffalo River Federal Navigation Channel. The contaminated sediments for the pilot scale demonstration came from the Buffalo River since this area had been sampled and tested in much more detail than the Buffalo Ship Canal, and, therefore it was anticipated that a sediment sample could be located and collected with greater assurance that it would contain significant concentrations of PAHs. It was desirable to treat more highly contaminated sediments during this demonstration since these are the sediments that are likely to be treated during any full scale remediation.

Sediments collected from the Buffalo River were used to evaluate the treatment technology rather than using sediments already deposited in the Corps of Engineers Confined Disposal Facility (CDF) No. 4 in Buffalo Harbor. Some of the PAHs contaminating the Buffalo River sediments are volatile and may escape the sediments when exposed to the atmosphere for significant lengths of time, or when they are rehandled several times, as sediments from the CDF would have been. Therefore, fresh sediments were collected for the pilot demonstration to ensure that significant amounts of the PAH contaminants did not volatilize prior to treatment.

The excavation of the contaminated sediments from the Buffalo River was accomplished by using floating plant consisting of a barge mounted crane and tug boat owned by Manson Construction Co. Inc. and under contract to the Corps of Engineers. An open clamshell bucket dredged approximately 15 cubic yards of sediments to be treated in the thermal desorption unit. Four to six feet of sediment was excavated in 10 to 12 feet of water at USEPA sample point 2501 and placed in four waste disposal bins (dumpsters) labeled "A" through "D" on the barge deck. Preliminary analytical results indicated that sediments at this location contained elevated levels of extractable residues from the surface of the sediments to 6 to 8 feet below the surface of the sediments.

## 2.6 SITE DESCRIPTION

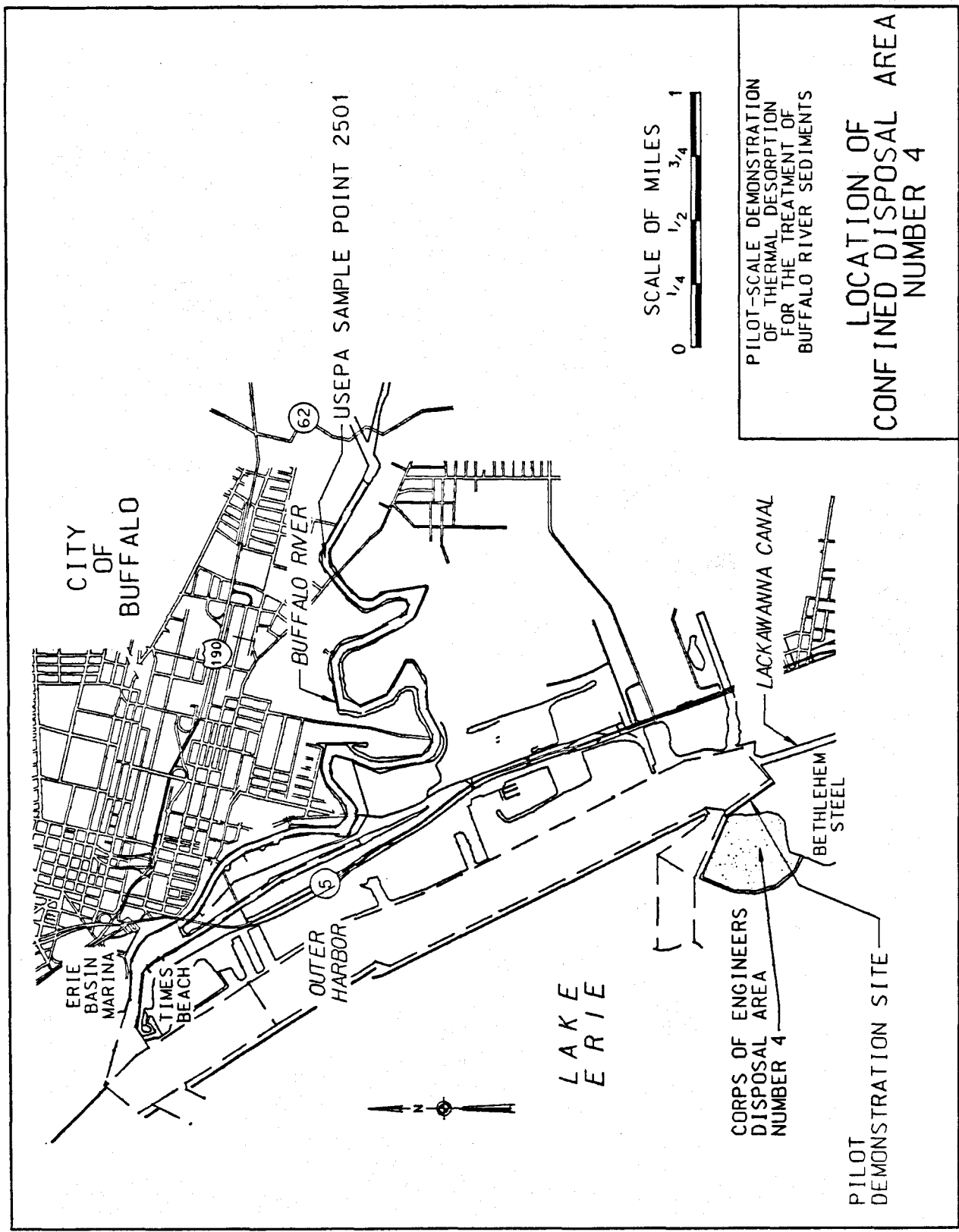
While other sites along the Buffalo River were considered, the demonstration was conducted within the confines of Buffalo District's CDF No. 4 due to several advantages of this site over privately owned or public lands that may have been available within the Area of Concern. Issues of liability and access were greatly simplified since the facility is owned by the Corps of Engineers. In addition, regulatory requirements had already been satisfied for using this facility to confine contaminated dredged sediments. The CDF had been designed and constructed in the mid 1970s to contain polluted sediments dredged from the Buffalo River and Buffalo Harbor. Community concerns would be eased if the demonstration were conducted within CDF No. 4 since it is located in an area removed from any housing or public access. Finally, the remoteness of the area minimized security problems.

Excavated sediments were transported by barge to CDF No. 4 (Figure 5). Contamination of the surrounding water bodies due to spillage was controlled by avoiding overloading the waste bins with sediment material. Transfer of the bins from the barge to the CDF was accomplished through the use of the barge mounted crane. The floating plant was secured in Buffalo Harbor, adjacent to the CDF, while the waste bins containing the dredged sediments were off-loaded onto Stoney Point Breakwater, the eastern boundary of the disposal facility (Figure 6).

CDF No. 4 is located at the southern end of the Buffalo Harbor, adjacent to the Bethlehem Steel Corporation's Lackawanna plant. Access to the CDF is available through the Bethlehem Steel plant. The pilot scale demonstration took place along the east side of the disposal facility, adjacent to the Stoney Point Breakwater (Figure 6). An existing access road along this portion of the dike was capable of carrying vehicular traffic, including tractor trailers loaded with demonstration equipment. Several acres were available within the disposal facility upon which the demonstration could be conducted. This included an area of 2 to 3 acres adjacent to the roughly 300-foot long pumpout pipe. This area was generally clear of trees and shrubs and was relatively level, though it did contain approximately 1 to 3 foot undulations in the surface contours.

### 2.6.1 Site Preparation

Prior to their mobile pilot scale unit arriving on site, Remediation Technologies, Inc. (RETEC) prepared the site. An area of roughly 10,000 square feet was cleared and prepared for the mobile thermal processor and support equipment which included: chiller, electric generator, water tank, inert gas tank (Nitrogen gas), office trailer, storage trailer, publicity tent, and drum storage area (Figure 7). Sufficient area was also available to provide parking for several vehicles. The demonstration area was constructed using a bulldozer to remove the surficial soils and stockpile them along



PILOT-SCALE DEMONSTRATION  
OF THERMAL DESORPTION  
FOR THE TREATMENT OF  
BUFFALO RIVER SEDIMENTS

# LOCATION OF CONFINED DISPOSAL AREA NUMBER 4

FIGURE 5

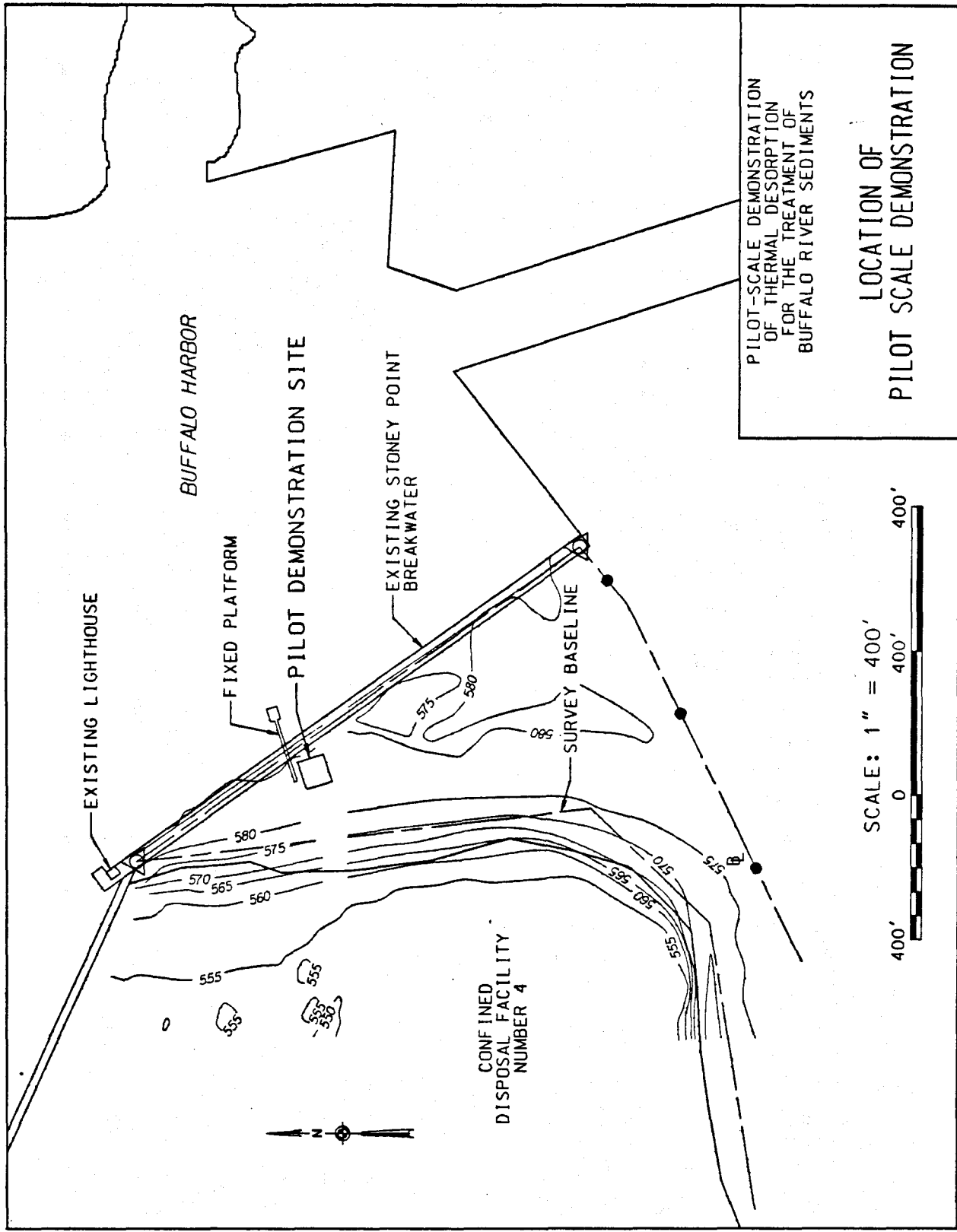


FIGURE 6

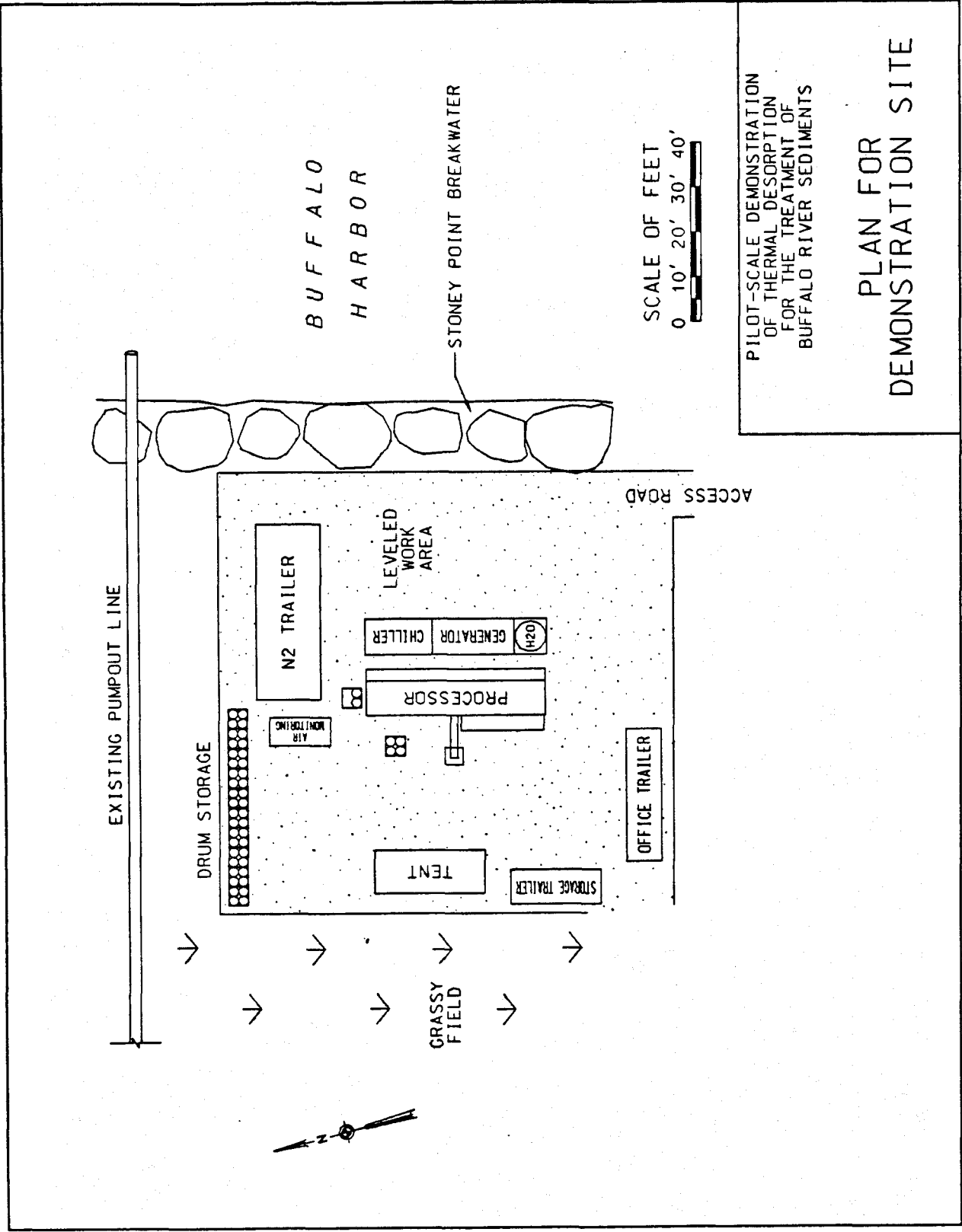


FIGURE 7

the western edge of the site. Approximately 300 tons of slag was applied to the cleared area to provide a firm working surface and improve water drainage. The slag material, purchased by Remediation Technologies, Inc. from the adjacent Bethlehem Steel plant, was spread with a bulldozer and compacted with a vibratory roller. RETEC performed site preparation and mobilized the pilot scale thermal desorption processor and support equipment to the site from October 7 through October 18, 1991.

## 2.7 MATERIAL HANDLING

### 2.7.1 Transport

The dredged sediments were placed into four dumpsters aboard the floating plant and transported to CDF No. 4 by Manson Construction Co. on October 7, 1991. Approximately 4 cubic yards of sediments were placed in each of four bins labeled "A" through "D" in order to track the sediments during the treatability study. Transfer of the dumpsters from the deck of the floating plant to CDF No. 4 was accomplished through the use of Manson's barge mounted crane. At this point, the sediments were turned over to the remediation contractor, RETEC, for pretreatment and treatment operations.

### 2.7.2 Screening

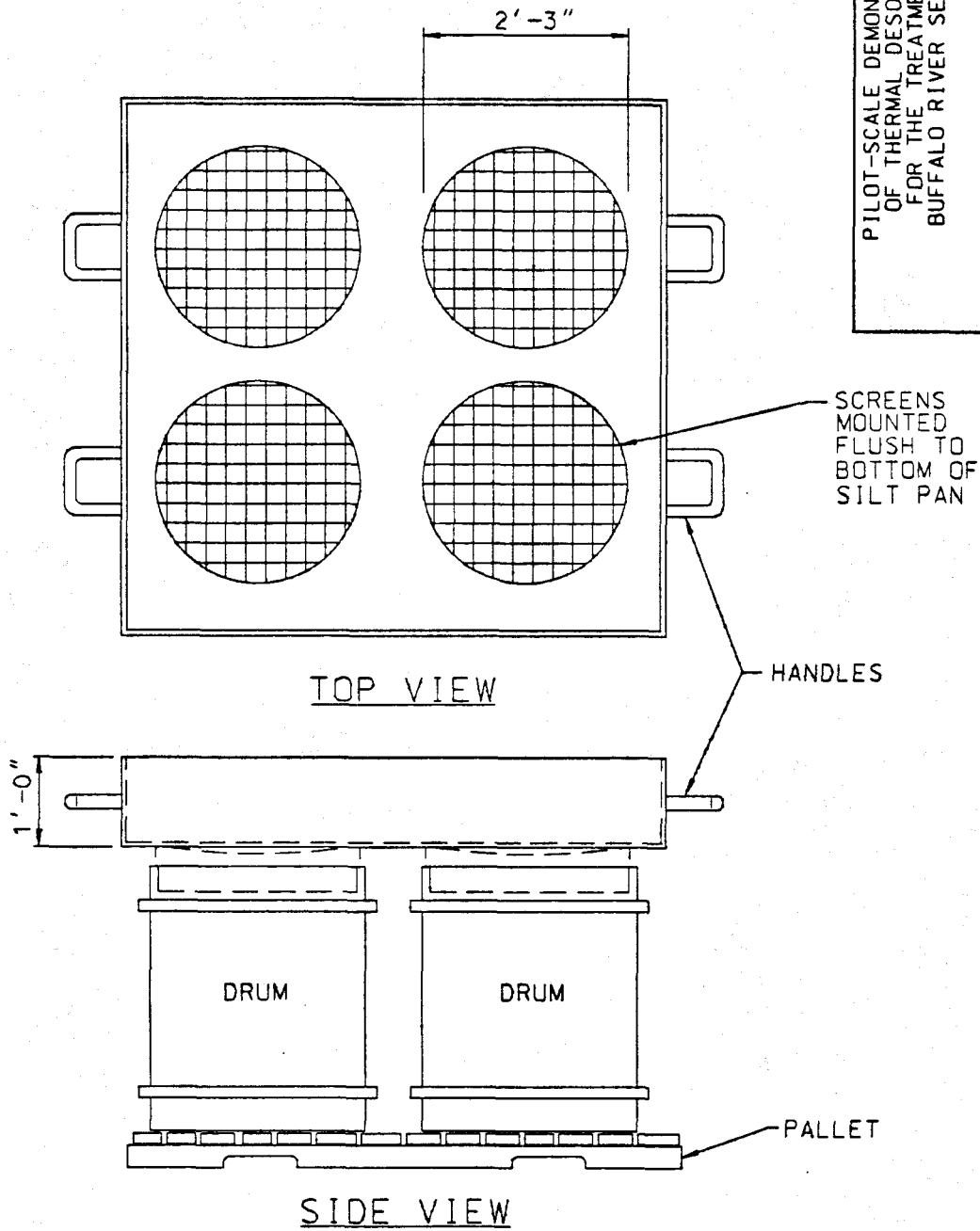
Prior to pilot scale treatment of the excavated sediment using the thermal desorption technology, it was necessary to remove particles and debris greater than 0.75 inches in size from the feed sediments. RETEC fabricated a sediment screening device which provided the capability of simultaneously filling four 55-gallon drums (Figure 8). The device covered an area 16 square feet in size and had four holes cut into it, each the size of a 55-gallon drum. An inflexible wire mesh was welded to each hole to screen objects greater than 0.75 inches in size. The screening device had 1 foot high walls and was capable of holding several cubic feet of material, the equivalent of a small backhoe bucket.

A backhoe was used to remove approximately 12 cubic yards of the sediments from the four dumpsters and place it in the screening device. Three cubic yards of sediment that could not be removed by backhoe were later removed by hand and disposed of in the CDF. The screening device was designed to allow undersized material to pass the wire mesh and fall into the 55-gallon drums by gravity. However, the cohesive nature of the sediments dredged from the Buffalo River for this demonstration prevented the material from passing through the screen by gravity. RETEC personnel then tried to use shovels to force the sediments through the screen with little success. They then used a backhoe bucket to force the sediments through the screen. This approach turned out to be time consuming and extremely inefficient. Screening by hand was the most efficient means found to screen the oversized material

PILOT-SCALE DEMONSTRATION  
OF THERMAL DESORPTION  
FOR THE TREATMENT OF  
BUFFALO RIVER SEDIMENTS

# SEDIMENT SCREENING DEVICE

FIGURE 8



from the sediments during the demonstration project. Each backhoe bucket full of sediments was handled by RETEC personnel outfitted in protective tyvek suits and gloves. Oversized material screened from the sediments consisted mostly of gravel and tree branches and amounted to well under 1 percent (by weight and volume) of the screened sediments. Oversized material was disposed of within the confined disposal facility while the screened material was stored in covered 55-gallon drums prior to being treated using the RETEC thermal desorption process.

#### 2.7.3 Storage

The 12 cubic yards of screened sediments were stored in 48 covered 55-gallon drums from the time they were screened, October 8 and 9, until the sediment was treated in the thermal desorption unit, between October 21 and November 25, 1991. Approximately 3 cubic yards of sediment were screened from each of the four bins (labeled A thru D) into twelve 55-gallon drums. The twelve drums from each bin of sediment were split into three groups of four drums each and labeled A1 (four drums), A2, A3, B1, B2, B3, C1, C2, C3, D1, D2, and D3.

#### 2.7.4 Addition of Water

Just prior to feeding the sediments for a particular run into the thermal unit, RETEC personnel mixed water with the four drums of sediment to be treated. The water content of the dredged sediments was relatively consistent, ranging from 40 to 45 percent. A relatively small amount of water, 2 to 3 percent, was added to the sediments from bin "A" in order to assist the feed operations by increasing the "pumpability" of the feed material. The water was added to each 55-gallon drum of sediment while the sediments were being mixed with an electric powered paddle mixer. The water content of the sediments from bins "B," "C," and "D" were similarly adjusted to approximately 45, 50, and 60 percent, respectively.

#### 2.7.5 Feed Operations

The normal material handling system for the thermal desorption unit was a bucket conveyor designed for bulk solid material, which appeared ineffective for feeding the high moisture, cohesive sediments dredged for the pilot scale demonstration. The contractor, RETEC, based this judgment on a simple field test, whether or not the sediments stuck to a spoon when the spoon was inverted. The Buffalo River sediments remained stuck to the spoon and, therefore, feeding the material into the thermal unit with the bucket conveyor was not attempted. For this demonstration, the sediments were fed into the processor by means of a diesel-powered peristaltic pump. The peristaltic pump was designed to provide a continuous and steady delivery of material to the thermal unit. The manufacturer described the pump as capable of processing up to 80 percent solids and particle sizes up to 0.75 inches in size. It was rated to have a



maximum delivery rate of 110 gallons per minute at a suction head of 29 feet. Initial project delays were encountered while waiting for site delivery of the pump from the manufacturer and in defining the proper material of construction for the peristaltic hose. A natural rubber material was finally selected on the basis of its resiliency. RETEC found the pump unsuccessful in pumping the screened sediments until a small amount of water was added as described above. Sediments were pumped into the thermal system just below the slide gate (air lock) on the processor to minimize infiltration of ambient air into the processing chamber.

## 2.8 THERMAL DESORPTION

Thermal desorption refers to the separation of contaminants from a solid matrix by heating to volatilize organic contaminants. The desorption process can be used in conjunction with separate processes, such as incineration, condensation, or adsorption, for subsequent control of the volatilized constituents. According to Remediation Technologies, Inc. the fact that, for some contaminants, efficient removals can be achieved at relatively low treatment temperatures makes thermal desorption a less costly approach than incineration for the remediation of solids contaminated with organic constituents. The desorption process is not effective in treating materials contaminated with inorganic contaminants.

The desorption process can be accomplished using various types of direct or indirect fired equipment. Applications using indirectly-fired methods are preferred in many cases since they generate a significantly smaller volume of off-gas than traditional drying or incineration systems. As a result, the capital and operating costs for the system can be significantly reduced.

### 2.8.1 System Description

Remediation Technologies, Inc. has developed a thermal desorption technology that reportedly has demonstrated applications as both a pre-treatment operation (dewatering, removal of volatile constituents) and final treatment operations for waste water treatment sludges from petroleum refineries as well as soils contaminated with organics (Remediation Technologies, Inc., 1992). RETEC's application of the technology relies on condensation to capture most of the organics volatilized by the thermal processor. Volatilized organics are condensed into a concentrated liquid stream which can subsequently be managed either on-site using further treatment systems or off-site at a permitted treatment/storage/disposal facility. The benefits of the system include lower capital costs relative to traditional thermal technologies, and permitting requirements that are less stringent than for incineration systems.

RETEC's system is based upon the use of an established, indirectly-heated thermal desorption/dryer system, the Holo-Flite Screw Processor, manufactured by the Denver Equipment Company, Colorado Springs, Colorado. The Holo-Flite processor is an indirect-heat exchanger commonly used to heat, cool, and dry bulk solids and slurries. The treatment system consists of a jacketed trough which houses a heated double-screw mechanism. The rotation of the screws promotes the forward movement of the material through the processor. The rotating augers are arranged in the trough so that the flights of the two screws mesh, facilitating the movement of material and improving the heat transfer.

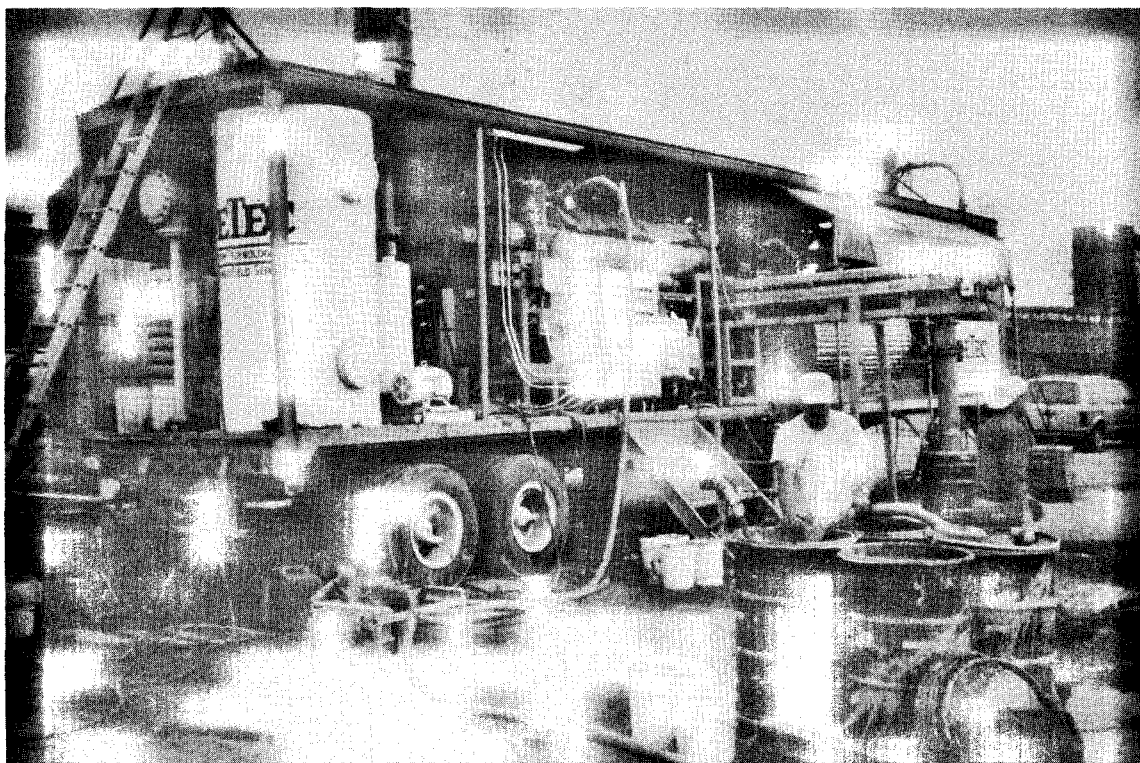
The RETEC processor uses a contained, non-contact circulating heat transfer media to elevate the temperature of the solids. The heated media continuously circulates through the hollow flights of the screw augers, travels the full length of the screws, and returns through the center of each shaft to the heater. The heat transfer fluid is also circulated through the trough jacket to provide additional heat transfer surfaces for improved volatilization. RETEC's system employs a unique heat transfer medium, a molten salt eutectic consisting of 53 percent potassium nitrate, 40 percent sodium nitrite, and 7 percent sodium nitrate. The use of this media provides the ability to achieve processing temperatures up to 850°F to effect appropriate removals of heavier organic species and increase the efficiency of the system in treating more complex solid matrices. In addition to the enhanced thermal properties, the salt eutectic provides significant safety benefits; the salt melt is non-combustible, it provides no risk of explosion, and potential vapors are non-toxic (Remediation Technologies, Inc., 1992). An inert atmosphere was maintained in the thermal treatment chamber through the controlled introduction of nitrogen gas to ensure that oxidation of the volatilized material did not occur.

Remediation Technologies, Inc. utilized its transportable demonstration system for the performance of this project (Figure 8A). The system, contained on a single 8-foot by 45-foot flat bed trailer, consisted of material feed equipment, thermal processor, indirect condensing system, and an activated carbon unit for the control of volatile organic constituents. A process flow diagram for this pilot scale system is shown in Figure 9. The design of the demonstration unit utilizes manual general control systems that are not equipped with feed interlocks.

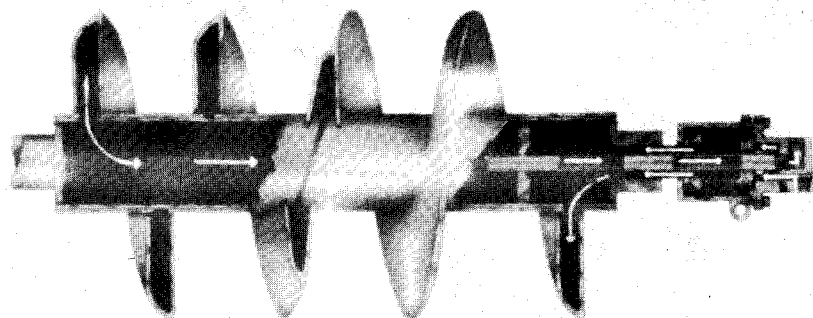
#### 2.8.1.1 Material Handling--

Generally, material to be processed by RETEC in their thermal desorption system is placed in a live bottom feed storage hopper with a 1.5 cubic yard capacity. The material is sized and conveyed to a bucket elevator using twin 6-inch diameter screws equipped with ribbon flights. The bucket elevator raises the material to a height of 17 feet to a feed auger which uses a single 6-inch ribbon flight screw to convey the material to the processor via a double slide gate (air lock). The slide gate is to prevent the leakage of ambient air into the processor.

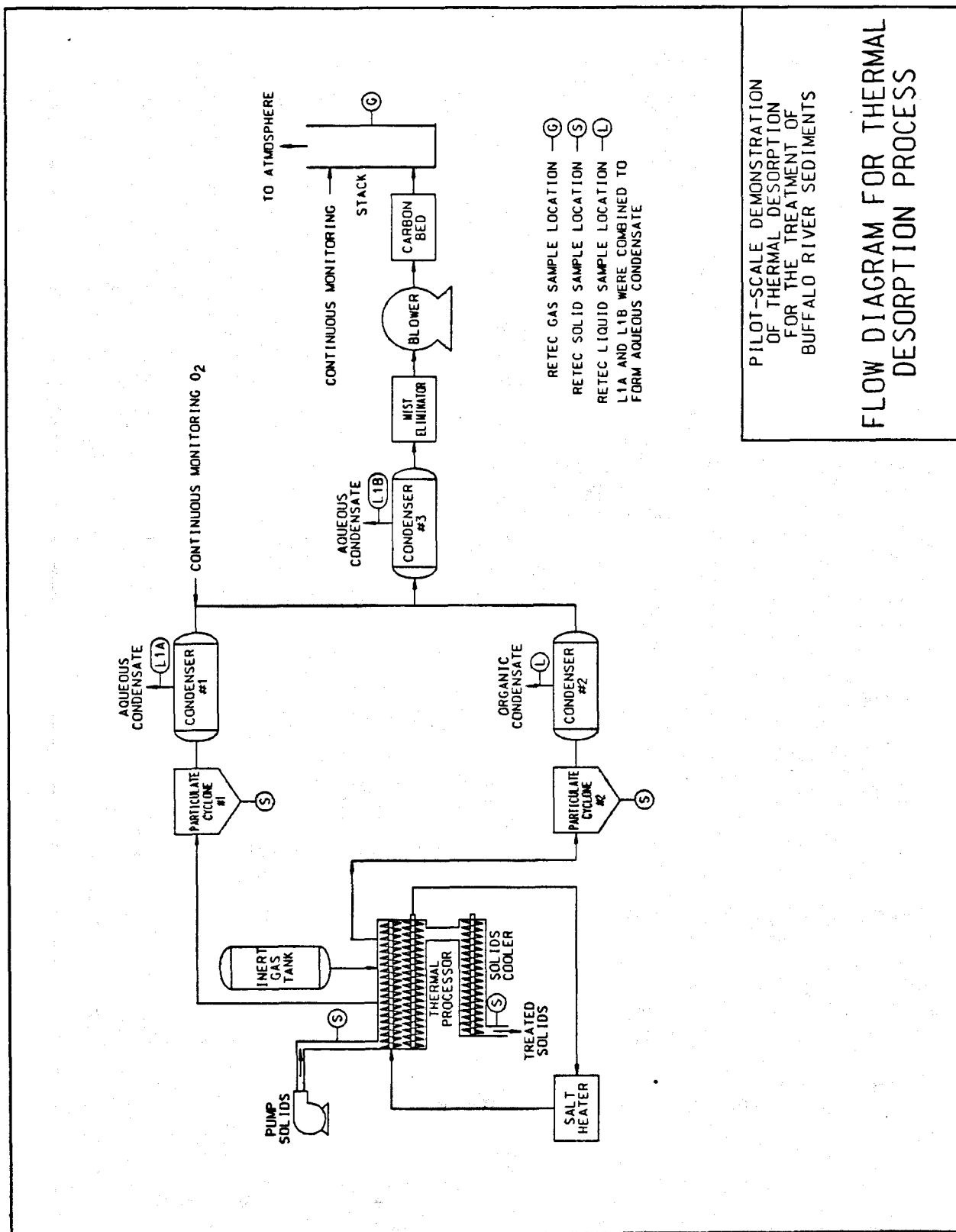
Figure 8A: Thermal desorption unit and screw processor



Thermal desorption unit at Buffalo River pilot demonstration site



Holo-flite screw processor



PILOT-SCALE DEMONSTRATION  
 OF THERMAL DESORPTION  
 FOR THE TREATMENT OF  
 BUFFALO RIVER SEDIMENTS

# FLOW DIAGRAM FOR THERMAL DESORPTION PROCESS

FIGURE 9

As stated previously, due to the cohesive nature of the Buffalo River sediments, a diesel powered peristaltic pump was used to deliver the screened Buffalo River sediments to the thermal processor during this demonstration project. The pump consisted of a 5 horsepower air cooled diesel engine attached to a high capacity peristaltic pump head. The flow rate of the pump was rated at 110 gallons per minute maximum. During this demonstration, the sediments were drawn through the pump at an average feed rate of 1 gallon per minute. A 2-inch diameter hose was used to draw the feed material from the 55 gallon drums and deliver them to the thermal processor. The pump discharge line was connected to the processor after the double slide gates (air lock).

#### 2.8.1.2 Thermal Processor--

The Holo-Flite thermal processor contained two 7-inch intermeshing screw conveyors and had the nominal capacity to treat 0.5 tons per hour of material (Figure 8A). The system was operated at media temperatures and solids residence times that allowed the solids to achieve temperatures in the range of 300 to 540°F. At these temperatures, organic constituents and moisture present in the waste material were volatilized and drawn away under negative pressure to the off-gas control system. Solids residence times in the processor were varied from 30 to 90 minutes through the use of a variable speed drive for the rotating augers.

The atmosphere in the treatment chamber was controlled during all treatment activities. The pressure inside the processor was maintained at -0.1 to -0.5 inches of water column and an "inert" atmosphere was maintained in the treatment chamber through the controlled introduction of nitrogen. RETEC used a commercially provided tank as the source of inert gas. The nitrogen ( $N_2$ ) gas was delivered to the processor at a flow rate of 5 to 30 cubic feet per minute. The oxygen content of the gas stream was monitored continuously during the operation of the treatment system to ensure that oxidation of the volatilized materials would not occur. Oxygen levels were consistently maintained below 17.5 percent.

Treated solids were fed by gravity to a second process auger designed to cool the solids prior to exiting the desorption unit. The "cooling screw" was also of the Holo-Flite design and used a single auger with chilled water as the cooling media. The cooling screw required approximately 12 gallons per minute (gpm) of water less than 90°F to cool the treated solids to a temperature of approximately 140°F. The temperature of the water was maintained using a closed-loop chiller system. The treated solids were discharged from the cooler through a rotary air lock into 55-gallon drums.

#### 2.8.1.3 Media Heater--

The salt eutectic was stored and heated in an enclosed, insulated stainless steel vessel having a capacity of approximately 600 gallons. The eutectic was heated electrically using 27 immersion heaters capable of providing one million BTU per hour of heating capacity to the unit and media temperatures of approximately 1,000°F. The heat medium was delivered to the thermal processor by means of a vertical pump with a submersible head. The pump had the capability to deliver up to 50 gpm of molten salt eutectic to the processor.

#### 2.8.1.4 Off-Gas Control--

The off-gas control system was designed to accommodate an off-gas flow rate of approximately 150 standard cubic feet per minute (scfm) and a "worst case" moisture and organic loading of 400 pounds per hour and 150 pounds per hour, respectively. Two particulate cyclones were used to remove any fine solid particles (greater than 10 microns) which may have been entrained with the off-gases. These solids were removed on a daily basis and combined with the treated solids for subsequent disposal. Two indirect heat exchangers, having a combined surface area of 200 square feet, were used to reduce the temperature of the gas leaving the processor to approximately 120°F and condense the majority of the entrained moisture and organics. An after cooler, condenser number 3, was placed in line to remove the remaining moisture and volatile organics from the off-gas stream (Figure 8). The exchanger was designed to achieve an exit gas temperature of 50°F. Cooling water was recirculated in a closed loop through a chiller having a capacity of 240,000 BTU per hour. Condensates were collected in two separate vessels prior to transfer from the system. The system was driven by a variable speed rotary blower capable of developing 300 scfm of flow at a vacuum of 3 inches of mercury.

The thermal system was equipped with an activated carbon system to control noncondensable organics prior to release to the atmosphere. The carbon system was charged with 1,500 pounds of carbon (Remediation Technologies, Inc., 1992). Volatile organic emissions from the system were monitored in the stack on a continuous basis .

#### 2.8.2 Pilot Scale Demonstration

Sediments from bins A and B were treated on-site in Buffalo, New York from October 21 through 31, 1991. Due to freezing temperatures and heavy snowfall in early November, the thermal desorption unit and its support equipment were transferred to RETEC's treatability laboratory in Acton, Massachusetts. Sediments from bins C and D were treated with the unit set up at this facility from November 18 through 25, 1991.

Remediation Technologies, Inc. monitored all pertinent process parameters at routine intervals during the demonstration. This approach was used to help optimize the removal efficiency of the thermal desorption demonstration unit as well as to develop data

for the design of full scale equipment. The data collected included material feed rate, revolutions per minute of the thermal processor augers, temperature of the heat transfer media entering and exiting the processor, residence time of the solid in the processor, temperature of solids entering and exiting the thermal processor, flow rate of the carrier gas, inlet temperature of the carrier gas, off-gas temperature, and mass rates of all process streams. Off-gas concentrations of oxygen and hydrocarbons were monitored continuously while other data was recorded at approximately 30 minute intervals during operation of the unit.

Process temperatures were monitored at 21 locations using thermocouples. Temperature signals were transmitted to a panel readout and subsequently recorded on field data sheets (Remediation Technologies, Inc., 1992). Gas pressures were monitored in the processing system using magnahelic gauges. Pressures were monitored within the headspace of the processor and across the principal components of the off-gas system to ensure proper operation of the system and to help anticipate maintenance problems such as poor heat transfer. The flow rate of inert gas into the processor was monitored by a standard flow meter while the off-gas flow rate from the thermal system was monitored in the off-gas stack by measuring the flow with a hot wire anemometer. The sediment feed rate to the processor was monitored by recording the volumetric displacement of the feed system per unit of time in conjunction with associated measurements of the density of the feed material. The accuracy of these observations was validated in the field using notations of the speed and capacities of the processing and cooling augers.

Upon review of the data RETEC determined that there was significant variability in both the pumping rate and density of feed material. Therefore, the average material feed rates were calculated by dividing the total mass of feed material for each test (weighted in the field) by the duration of time that material was fed into the processor.

#### 2.8.2.1 Sediment A--

Sediment from bin A was treated in the thermal desorption unit from October 21 thru October 24, 1991. The material was processed after mixing with a small amount of Buffalo Harbor water (2% to 3%) to improve the "pumpability" of the material. The feed rate and residence time were varied for the feed material designated A1, A2, and A3. Tables 7A and 7B present the process data for each treatment run as recorded by RETEC. Sediment from the three runs, A1, A2, and A3, was treated at feed rates ranging from 346 to 716 pounds per hour (wet weight) and residence times ranging from 30 to 90 minutes. The process temperatures ranged from 933°F to 938°F for the heat transfer media entering the processor, 902°F to 911°F for the heat transfer fluid leaving the processor, and 380°F to 535°F for the treated solids exiting the processor.

TABLE 7A  
PROCESS PARAMETER VALUES AS MEASURED BY RETEC, INC.  
(English Units)

PARAMETER	Run											
	A1	A2	A3	B1	B2	B3	C1	C2	C3	D1	D2	D3
Date (1991)	10/21	10/23	10/24	10/25	10/30	10/31	11/18	11/19	11/20	11/21	11/22	11/25
Residence Time (min)	90	60	30	45	60	90	90	60	45	90	60	45
Feed Rate (lbs/hr)	346	502	716	860	644	334	484	635	862	388	575	724
Temperatures (°F)												
Heat Transfer Media In	938	937	933	937	962	951	921	832	834	888	872	861
Heat Transfer Media Out	911	905	902	908	929	920	881	804	802	853	843	824
Sediment In	60	60	60	60	60	60	60	60	60	60	60	60
Sediment Out	535	480	380	300	362	344	392	415	474	367	423	303
Inert Off-Gas	917	950	860	230	656	756	854	974	900	977	965	975
Heat Input (BTU/hr)	367,390	435,420	421,820	421,820	449,030	421,800	554,280	381,000	435,000	476,250	394,600	503,460

TABLE 7B  
PROCESS PARAMETER VALUES AS MEASURED BY RETEC, INC.  
(SI Units)

PARAMETER	Run											
	A1	A2	A3	B1	B2	B3	C1	C2	C3	D1	D2	D3
Date (1991)	10/21	10/23	10/24	10/25	10/30	10/31	11/18	11/19	11/20	11/21	11/22	11/25
Residence Time (min)	90	60	30	45	60	90	90	60	45	90	60	45
Feed Rate (kg/hr)*	157	228	325	390	292	151	220	288	391	176	261	328
Temperatures (°C)*												
Heat Transfer Media In	503	503	501	502	517	511	494	444	446	476	467	461
Heat Transfer Media Out	488	485	483	487	498	493	472	429	428	456	451	440
Sediment In	16	16	16	16	16	16	16	16	16	16	16	16
Sediment Out	279	249	193	149	183	173	200	213	246	186	217	151
Inert Off-Gas	492	510	460	110	347	402	457	523	482	525	518	524

\* These data are converted from English Units and are rounded to the nearest whole number.  
DATA SOURCE: Referenced RETEC, Inc. Report



Some operational problems were encountered during the processing of the sediment. Initial feed to the unit was erratic due to RETEC's unfamiliarity with the peristaltic pump and because the pump was delivered from the manufacturer with a peristaltic hose that was not compatible with the sediment material (Remediation Technologies, Inc., 1992). A consistent flow was established once the pump hose was changed from a high density polyethylene to a natural rubber material. In addition, some fouling of the processor was encountered between the processing of samples A2 and A3. RETEC reported that the fouling was the result of caking of dried sediments around the processing augers. The processor was partially disassembled and the caked solids were manually removed from around the augers. The buildup of dried material was thought to have been due to the cohesive nature of the fine grained sediments fed into the processor.

#### 2.8.2.2 Sediment B--

Material from bin B was treated in the thermal desorption unit from October 25 thru October 31, 1991. The sediment was processed after mixing with Buffalo Harbor water to achieve a target moisture content of 45 percent prior to treatment. The feed rate and residence time were varied for the feed material designated B1, B2, and B3. Tables 7A and 7B present the process data for each treatment run as recorded by RETEC. Sediment from the three runs, designated B1, B2, and B3, was treated at feed rates ranging from 334 to 860 pounds per hour (wet weight) and process residence times ranging from 45 to 90 minutes. The process temperatures ranged from 937°F to 962°F for the heat transfer media entering the processor, 908°F to 929°F for the heat transfer fluid leaving the processor, and 300°F to 362°F for treated solids exiting the processor, as measured by RETEC personnel.

As in the case of the treatment of sediment from bin A, there was a significant buildup of dried material in the thermal processor. While treating sediment sample B3 the buildup became so severe that the processing augers could no longer rotate and a shear pin on the auger system broke. The thermal system was disassembled, the caked solids were manually cleaned out, and the shear pin was replaced.

#### 2.8.2.3 Sediment C--

On November 3 and 4, 1991 a snow storm left approximately 1 foot of snow covering the project area and the air temperature dropped to between 25 and 30°F. The unprocessed Buffalo River sediments from bins C and D as well as hydraulic lines on the thermal desorption unit froze. This made it impossible to operate the thermal desorption unit or to feed the sediments through the peristaltic pump into the processor until warmer temperatures returned. Rather than waiting for warmer temperatures to return, the Corps of Engineers and Remediation Technologies, Inc. agreed that the pilot scale demonstration should be completed at RETEC's indoor treatability facility in

Acton, Massachusetts. During the weeks of November 4 and November 11, 1991, RETEC personnel demobilized the pilot scale unit and set the unit up in their Acton facility. They also transported the untreated sediments from bins C and D from Buffalo, New York to Acton, Massachusetts.

Sediments from bin C were processed through the thermal desorption unit on November 18, 19, and 20, 1991. Tap water was mixed with the sediments to achieve a target moisture content of approximately 50 percent prior to treatment. The feed rate and residence time of the sediment in the thermal unit were varied for the material designated as C1, C2, and C3. Tables 7A and 7B present the processed data for each treatment run as recorded by RETEC. Sediment from the three runs, C1, C2, and C3, was treated at feed rates ranging from 484 to 862 pounds per hour (wet weight) and process residence times ranging from 45 to 90 minutes. The process temperatures ranged from 832°F to 921°F for the heat transfer media entering the processor, 802°F to 881°F for the heat transfer fluid leaving the processor and 392°F to 474°F for treated solids exiting the processor.

#### 2.8.2.4 Sediment D--

Sediment from bin D was treated in the thermal unit November 21, 22, and 25, 1991 in Acton, Massachusetts. Tap water was mixed with the sediments to achieve a moisture content of approximately 60 percent prior to treatment. The feed rate and residence time of the sediment in the thermal unit were varied for the process runs using sediment samples D1, D2, and D3. Tables 7A and 7B present the process data for each treatment run as recorded by RETEC. Sediment from the three runs was treated at feed rates ranging from 388 to 724 pounds per hour (wet weight). Process temperatures ranged from 861°F to 888°F for the heat transfer media entering the unit, 824°F to 853°F for the heat transfer fluid leaving the processor, and 303°F to 423°F for the treated solids exiting the processor.

## 2.9 RESIDUALS MANAGEMENT

Most of the residuals from the pilot scale demonstration were disposed of by Remediation Technologies, Inc. While operating in Buffalo, New York, residual solids and liquids from the bin A and B treated sediments were collected in 55-gallon drums. Treated solids from the sediment A1 run were disposed within the Corps Confined Disposal Facility. The remaining treated solids, including the cyclone material, were used in a solidification/stabilization demonstration. RETEC sealed the organic condensate in 55-gallon drums and transported it to Acton, Massachusetts for proper disposal when the thermal unit was demobilized from Buffalo, New York. The aqueous condensate was emptied from the 55-gallon drums into the confines of Confined Disposal Facility Number 4.

All residuals from the treatment of sediments from bins C and D, as well as the organic condensate from the A and B process runs performed in Buffalo, were disposed of by RETEC at a licensed disposal facility in the New England region.

## 2.10 SOLIDIFICATION OF SOLID RESIDUE

A portion of the residual solids from the treatment process were solidified/stabilized. The solidification/stabilization technology immobilizes certain contaminants by binding them into a concrete-like, leach resistant mass. The formation of the solidified product is achieved during a hydration reaction in which free water is bound to the setting agent. The physical and chemical stability of the resulting product are functions of the sediment/residual characteristics, type of setting agent, and additives used. Cement processes reduce the mobility of heavy metals due to their conversion to insoluble hydroxides or carbonates because of the elevated pH of cement.

Under the ARCS Program, the Corps of Engineers evaluated solidification/stabilization at bench-scale for potential treatment of contaminated sediments from the Buffalo River. The evaluation was conducted to determine whether physical and chemical properties of the sediment would be improved. Results are reported in "An Evaluation of Solidification/Stabilization Technology for Buffalo River Sediments" (Fleming, Averett, Chennell, Perry, 1991) and are summarized here. Based on analyses of the untreated sediment, five metals were selected for evaluation: chromium, copper, lead, nickel, and zinc.

Initial screening tests for the laboratory were conducted on the sediment to narrow the range of binder-to-soil ratios to be prepared in the detailed evaluation. Three binder materials were evaluated: cement, kiln dust, and lime/fly ash. Based on the results of the initial screening tests, binder-to-soil ratios were selected for the detailed evaluation. Specimens were prepared by mixing sediment and binder materials and molding the mixture. The specimens were cured for 28 days at 23°C and 98-percent relative humidity.

Physical tests including unconfined compressive strength (UCS), freeze/thaw durability, and wet/dry durability were run to determine if the physical handling properties of the sediment were improved. Contaminant release tests were conducted to determine the effectiveness of the binder materials on immobilization of the contaminants. Based on the results of the UCS tests, specimens were selected for evaluation of contaminant release properties. The solidification/stabilization specimens were subjected to the U.S. Army Engineers Waterways Experiment Station serial leach test and the toxicity characteristic

leaching procedure (TCLP). The serial leach test results were compared to the drinking water standards, and the TCLP results were compared to the regulatory thresholds.

Based on the TCLP results for crushed specimens, the cement and kiln dust solidification/stabilization processes were effective in reducing the leachability for lead, nickel, and zinc. Leachability of copper and chromium was increased by the processes when compared with untreated sediment for both the TCLP and the serial leach test. Heavy metal releases from test specimens may have been increased during the tests by the destruction of the physical integrity of the specimens. If physical stabilization of Buffalo River sediment is to be performed, cement was recommended as the appropriate binder on the basis of strength, durability, and leachability.

As a result of this investigation it was decided that a Type I Portland cement would be used for the pilot scale demonstration and that a binder (cement) to treated solids ratio of from 0.1 to 0.6 would be used. The solidification/stabilization tests were initiated on October 30 and November 1, 1991. The treated solids from process runs B1 and B3 were combined and mixed with Type I Portland cement to achieve a cement to treated solids ratio of approximately 0.1. A total of 110 pounds of cement were mixed with 1,000 pounds of treated solids and 380 pounds of water in two batches in a 9 cubic foot gas powered concrete mixer (Table 8). The resulting mass was placed and vibrated in a 3-foot diameter sonotube (mold) form for curing. The form was set on plastic sheeting prior to placement while the top of the sonotube was covered with plastic sheeting shortly after placement of the mix.

TABLE 8  
SOLIDIFICATION/STABILIZATION MIXES

Nominal Cement to Treated Solids Ratio	Source of Treated Solids (Process Run)	Pounds of Material			
		Treated Solids	Cement	Water	Total
0.1	B1, B3	1000	110	380	1490
0.2	A2	900	179	355	1434
0.4	A3	950	376	495	1821
0.6	B2	1036	619	545	2200

The treated solids from process run A2 were mixed with Portland cement to achieve a cement to treated solids ratio of approximately 0.2. A total of 179 pounds of cement were mixed with 900 pounds of treated solids and 355 pounds of water in two batches in the concrete mixer. Solids resulting from the processing of A3 sediment were mixed with cement to achieve a cement to treated solids ratio of approximately 0.4. Three hundred seventy six pounds of cement were mixed with 950 pounds of treated solids and 495 pounds of water in two batches in the concrete mixer. Residual solids from the processing of B2 sediments were mixed with Portland cement to achieve a cement to treated solids ratio of approximately 0.6. Six hundred nineteen pounds of cement were mixed with 1,036 pounds of treated solids and 545 pounds of water in two batches. In each case the resulting mass was placed and vibrated in a sonotube form for curing in a manner similar to that described in the above paragraph. The plastic sheeting was removed from the top and the sonotube form was stripped from the four solidified masses 5 to 7 days after mixing operations. The plastic sheeting was then placed on top of the four masses and later removed after an additional four weeks of curing in the field.

## 2.11 EXECUTION AND COSTS

Sediments were dredged from the upper Buffalo River with a barge mounted crane and transported to CDF No. 4 by Manson Construction Co. on October 7, 1991. Sediments were screened through a 0.75 inch wire mesh on October 8 and 9 by RETEC personnel to remove oversized material. The screened sediments were then stored in covered 55 gallon drums while RETEC performed site preparations and mobilized the pilot scale thermal desorption processor and support equipment to the site from October 7 through October 18, 1991. Startup of the desorption unit occurred on October 21 with sediments being treated on a 5 days per week, one shift per day basis through October 31. In early November, freezing temperatures and heavy snowfall necessitated that the treatment unit and its support equipment be transferred to RETEC's treatability laboratory in Acton, Massachusetts. The remaining sediments were treated on a one shift per day, 5 days per week basis with the unit set up at this facility from November 18 through 25, 1991.

Costs for the entire project were estimated in the work plan to be \$665,500, including the cost of project management, preparation of a sampling and analysis plan and health and safety plan, site preparation, sediment excavation and remediation, project monitoring, including extensive sampling, sample analysis, and preparation of this report. Actual cost of the demonstration, shown in Table 9, was approximately \$636,000. It should be noted that laboratory analytical work performed on samples collected during the demonstration cost more than the actual remediation of the sediments. This is not uncommon for a pilot scale demonstration of this nature.

TABLE 9  
COST OF THERMAL DESORPTION PILOT SCALE DEMONSTRATION

Activity	Cost
Project Management	\$75,000
Health and Safety Plan	5,000
Sampling and Analysis Plan	15,000
Sediment Excavation (Incl. Misc. Equip. & Supplies)	15,000
Site Preparation	23,100
Thermal Desorption Demonstration	173,000
Demonstration Monitoring/Sample Collection (Incl. Stack Gas Monitoring)	65,000
Sample Analysis	225,000
Solidification/Stabilization	5,000
Report Preparation	<u>35,000</u>
Total	\$636,100

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## 2.12 MONITORING

### 2.12.1 Process Monitoring by Remediation Technologies, Inc. (RETEC)

Evaluation of this project was conducted by performing detailed characterization of the contaminated sediment. Sediment collected from the river was sampled as soon as practical after placement in the four bins to provide an initial determination of the organics and heavy metals in the sediment. A second series of samples was collected following transport and screening to remove oversized material in order to evaluate losses subsequent to the sediments being dredged. Material passing the screen was stored in barrels and became the feed to the thermal processor. A third set of samples was collected just prior to the material being fed to the processor. Treated solids discharged from the processor were sampled for comparison to the feed and determination of the efficiency of the thermal process in removing contaminants of concern. Other process residuals were also characterized to evaluate contaminant losses from the overall process.

The critical contaminants for the evaluation were PAHs. Other organic compounds, including PCBs, were also present. Heavy metals were important contaminants in the sediment, but their concentrations were not expected to be significantly affected by the pretreatment and treatment processes. The effectiveness of the solidification/stabilization process was evaluated on the basis of changes in leachate quality for the solidified material compared to the treated solids.

Sediments from the four bins were managed to produce a range of water contents for the Buffalo River sediment fed to the thermal processor. One bin of sediments was maintained close to the water content of the as-dredged sediment and was the first material fed to the processor. For barrels of sediment from the three remaining bins, successively increasing amounts of water were added to the feed. Three operating conditions were run for each water content, i.e., for each bin of sediment, yielding a total of 12 runs for the pilot project.

The pilot scale demonstration was conducted by the USEPA's Great Lakes National Program Office. The U.S. Army Corps of Engineers, Buffalo District acted as project manager on this demonstration in support of the USEPA. The District was responsible for supervision of the project, including coordination with contractors, and oversight of the field demonstration, including field sampling. The Corps of Engineers, Waterways Experiment Station provided technical support to the Buffalo District in field sampling activities and implementation of the Quality Assurance Project Plan. Laboratory analytical work was performed by Battelle Marine Science Laboratory. RETEC was responsible for collection and recording of all operational data (U.S. Army Engineer Waterways Experiment Station, 1991).

Remediation Technologies, Inc. monitored all pertinent process parameters at routine intervals during the demonstration. Regular monitoring was done to help optimize the removal efficiency of the thermal desorption demonstration unit and to develop data for full scale design. The data collected included feed rate, revolutions per minute of the thermal processor augers, input and output temperatures of the heat transfer media, residence time of the solids in the processor, temperature of solids entering and exiting the thermal processor, carrier gas flow rate, carrier gas inlet temperature, off-gas temperature, and mass rates of principal process streams. Off-gas concentrations of oxygen and hydrocarbons were monitored continuously, while other data was recorded at approximately 30 minute intervals during operation. A complete presentation of RETEC's methods and results are given in: "Field Demonstration of RETEC Thermal Unit for Remediation of Buffalo River Sediments, Buffalo River Area of Concern," RETEC, Inc., March 1992.

In summary, process temperatures were monitored at 21 locations using thermocouples. Temperature signals were transmitted to a panel readout and subsequently recorded on field data sheets (Remediation Technologies, Inc., 1992). Gas pressures were measured in the processing system with magnahelic gauges. Pressures were monitored within the processor headspace and across the principal components of the off-gas system to ensure proper operation of the system and to help anticipate maintenance problems such as poor heat transfer. Inert gas flow rate into the processor was monitored by standard flow meter while flow rate in the off-gas stack was measured with a hot wire anemometer. The sediment feed rate to the processor was determined by recording the volumetric displacement of the feed system per unit time and converting to mass flow rate using the measured density of the feed material. The validity of these observations was assessed by comparing them against known capacities of the processing and cooling augers at the associated operating conditions. Based on these data, RETEC determined that there was significant variability in both the pumping rate and density of feed material. Therefore, average material feed rates were calculated for each run by dividing the total mass of feed material by the feed duration.

## 2.12.2 Air Monitoring

### 2.12.2.1 Air Monitoring by Remediation Technologies, Inc. (RETEC) --

RETEC, Inc. monitored process exhaust gas for total hydrocarbon content and for concentration of four specific volatile hydrocarbons. These measurements were performed as an instantaneous assessment of process performance, and were used with moisture data to optimize process efficiency. Total hydrocarbon concentration was measured using a flame ionization detector and a propane standard. This result was converted to an emissions rate using the stack gas flow rate. Overall emissions ranged from zero to 1178 ppm as propane, with an average of 10 ppm as propane. The average discharge rate was 0.02 pounds per hour. Average emissions rates represent the total organic compound emissions for processing Buffalo River sediment under the operating conditions of the pilot demonstration.

Additionally, specific analyses were performed for benzene, toluene, ethylbenzene, and xylene (BTEX) using a modified NIOSH procedure employing activated charcoal tubes. Exhaust concentrations of benzene averaged 0.61 mg per cubic meter (0.137 grams per hour). Concentrations of ethylbenzene, toluene, and xylene averaged 0.06, 0.02, and 0.02 mg per cubic meter, respectively (Table 10). Again, these average rates represent expected BTEX emissions for thermal desorption of Buffalo River sediment under conditions of periodic optimization.

Details of RETEC's air sampling procedures and their use in process optimization are given in "Field Demonstration of RETEC Thermal Unit for Remediation of Buffalo River Sediments, Buffalo River Area of Concern," RETEC, Inc., March 1992.



TABLE 10  
RESULTS OF VAPOR MONITORING BY RETEC, INC.

PARAMETERS	Date: 10/24/91	mg/m <sup>3</sup>	g/hr	mg/m <sup>3</sup>	g/hr	Date: 10/30/91	mg/m <sup>3</sup>	g/hr	mg/m <sup>3</sup>	g/hr	Date: 10/31/91	mg/m <sup>3</sup>	g/hr	mg/m <sup>3</sup>	g/hr	Date: 11/21/91	mg/m <sup>3</sup>	g/hr	mg/m <sup>3</sup>	g/hr	Date: 11/22/91	mg/m <sup>3</sup>	g/hr	mg/m <sup>3</sup>	g/hr	Date: 11/25/91	mg/m <sup>3</sup>	g/hr
Benzene		1.4	0.310	0.57	0.126		0.52	0.115	0.44	.098		0.55	0.122	0.23	0.051		0.09	0.199										
Ethylbenzene		0.04	0.003	NA	NA		NA	NA	NA	NA		NA	NA	NA	NA		NA	NA										
Toluene		NA	NA	NA	NA		NA	NA	NA	NA		0.13	0.028	NA	NA		NA	NA										
Xylene		0.15	0.033	NA	NA		NA	NA	NA	NA		NA	NA	NA	NA		NA	NA										
Total BTEX		1.6	0.354	NA	NA		NA	NA	NA	NA		NA	NA	NA	NA		NA	NA										

NA = Not Analyzed

DATA SOURCE: Referenced RETEC, Inc. Report: Results are for 8 hour time integrated samples averaged over 8 hour gas flow rate or 8 hour time period as indicated.

#### 2.12.2.2 Air Monitoring by E-Three, Inc.--

E-Three, Inc. sampled stack emissions for concentration of total particulates, polychlorinated biphenyls (PCBs), and polycyclic aromatic hydrocarbons (PAHs). Sampling was performed in several 2-3 hour increments over a period of 10 days. Data generated by E-Three, Inc. was not used for on-site process optimization. Rather, the E-Three data was used to quantify emissions of semi volatiles and particulates for the process conditions used by RETEC. Sampling and analytical methods numbers are presented in Table 11, and a summary of analytical results is given in Table 12. Results are for time integrated samples taken over 3 hours and averaged over the number of dry standard cubic meters (DSCM) of air discharged from the system in that time. While these emissions are referred to as "air emissions," the actual composition is not strictly that expected for air. The processing system is blanketed with nitrogen to prevent combustion of organic constituents in the sediment, and the resulting emissions are 7 percent oxygen. Complete test results are available in "Stack Emission Test Report: Demonstration Thermal Desorption Project, Buffalo River Sediments," E-Three, Inc., Buffalo, NY.

#### 2.12.3 Corps of Engineers Monitoring

The Corps of Engineers performed general project oversight including direction of all contractors involved in field operations. Additionally, the Corps of Engineers conducted an intensive sampling and analysis program as described below and as delineated in the referenced Quality Assurance Project Plan. Results of this program are summarized in the following text, and the complete data set for this effort is presented in Appendix B. Process data was provided by RETEC, Inc., and appears previously in Table 7A and 7B.

##### 2.12.3.1 Sampling--

The Corps of Engineers sampled solid and liquid streams both into and out of each process step and submitted these samples to Battelle Laboratories for analysis. Analytical methods employed for these analyses are given in Table 13 for sediments and in Table 14 for water samples (U.S. Army Engineer Waterways Experiment Station, 1991). The primary intent of this sampling and analysis program was to determine process stream changes, and therefore remediation effectiveness, at each stage of the pilot operation. Other goals for sampling and analysis were to (a) obtain information for process scaleup, (b) assess reductions in contaminant concentrations of the sediment during remediation, and (c) assess effectiveness of cementitious stabilization as a treatment for process residuals. A process flow diagram depicting Corps of Engineers sampling points is given in Figure 10.

TABLE 11

SUMMARY OF AIR SAMPLING AND ANALYTICAL PROCEDURES:  
E-THREE/BATTELLE

Parameter	Sampling Method	Analytical Method
Particulates	EPA Method 5	Gravimetric
PCB/PAH	EPA Method 23	High Resolution GC/MS
CO <sub>2</sub> , O <sub>2</sub> , N <sub>2</sub>	EPA Methods 3 and 3A	Orsat/CEM
CO	EPA Method 10	GFC-NDIR
Moisture	EPA Method 4	
Volumetric Flow	EPA Method 1 & 2	
Dioxins	EPA Method 23	High Resolution GC/MS

DATA SOURCE: Referenced E-Three, Inc. Report

TABLE 12A  
AIR EMISSIONS OF POLYCHLORINATED BIPHENYLS  
IN MICROGRAMS PER DRY STANDARD CUBIC METER

PCB Homolog	Minimum Emissions ug/DSCM @ 7% O <sub>2</sub>	Maximum Emissions ug/DSCM @ 7% O <sub>2</sub>
Monochlorobiphenyl	<0.33	<1.81
Dichlorobiphenyl	<0.33	<1.81
Trichlorobiphenyl	<0.33	<1.81
Tetrachlorobiphenyl	<0.66	<3.62
Pentachlorobiphenyl	<0.66	<3.62
Hexachlorobiphenyl	<0.66	<3.62
Heptachlorobiphenyl	<1.01	<5.55
Octachlorobiphenyl	<1.01	<5.55
Nonachlorobiphenyl	<1.01	<5.55
Decachlorobiphenyl	<1.67	<9.18

These values are all below detection limits. Detection limits are varying due to (a) compound - specific nature of detection limits and (b) solids content of the sample.

TABLE 12B  
AIR EMISSIONS OF PARTICULATES IN MICROGRAMS  
PER DRY STANDARD CUBIC METER

	Minimum Emissions ug/DSCM @ 7% O <sub>2</sub>	Maximum Emissions ug/DSCM @ 7% O <sub>2</sub>
Particulates	0.0019	0.0027

TABLE 12C  
AIR EMISSIONS OF POLYCYCLIC AROMATIC HYDROCARBONS  
IN MICROGRAMS PER DRY STANDARD CUBIC METER

PAH	Minimum Emissions ug/DSCM @ 7% O <sub>2</sub>	Maximum Emissions ug/DSCM @ 7% O <sub>2</sub>	Average Emissions ug/DSCM @ 7% O <sub>2</sub>
Naphtahalene	27.78	322.98	114.43
Acenaphthylene	0.25	3.34	1.31
Acenaphthene	0.78	6.61	3.13
Fluorene	1.23	3.04	4.36
Phenanthrene	8.68	129.62	42.96
Anthracene	0.84	12.89	4.61
Fluoranthene	3.16	93.26	27.14
Pyrene	1.81	34.67	13.62
Benz(a)anthracene	0.36	24.30	9.05
Chrysene	0.43	10.60	3.85
Benzo(bjk)fluoranthrene	0.85	19.45	6.01
Benzo(a)pyrene	0.21	2.42	1.12
Indo(1,2,3-cd)pyrene	0.21	3.66	1.40
Dibenz(a,h)anthracene	0.20	0.32	1.65
Benzo(g,h,i)perylene	0.23	1.45	0.80
TOTAL			235.44

TABLE 12D  
AIR EMISSIONS OF DIOXINS IN MICROGRAMS  
PER DRY STANDARD CUBIC METER

Dioxin	Minimum Emissions ng/DSCM @ 7% O <sub>2</sub>	Maximum Emissions ng/DSCM @ 7% O <sub>2</sub>
2378-TCDD	0.03480	0.25800
12378-PeCDD	0.00727	0.26100
123478-HxCDD	0.00220	0.00347
123678-HxCDD	0.00304	0.00313
123789-HxCDD	0.00573	0.00318
1234678-HpCDD	0.00156	0.00373
OCDD	0.00122	0.00115

Emissions reported as 2378-TCDD Toxicity Equivalents (i.e., they are quantitated based on a 2378-TCDD Standard)

TABLE 12E  
AIR EMISSIONS OF FURANS IN MICROGRAMS  
PER DRY STANDARD CUBIC METER

Furan	Minimum Emissions ng/DSCM @ 7% O <sub>2</sub>	Maximum Emissions ng/DSCM @ 7% O <sub>2</sub>
2378-TCDF	0.0000	0.0013
12378-PeCDF	0.0000	0.0000
23478-PeCDF	0.0000	0.3530
123478-HxCDF	0.0000	0.0838
123678-HxCDF	0.0003	0.0686
234678-HxCDF	0.0000	0.0015
123789-HxCDF	0.0000	0.0217
1234678-HpCDF	0.0000	0.0237
1234789-HpCDF	0.0000	0.0000
OCDF	0.0000	0.0000

\* Emissions reported as 2378-TCDD Toxicity Equivalents  
(EPA 1989)

Zero values in Table 12E were the result of taking total weight of the compound collected on resin over the 2-3 hour sample period and dividing by the dry standard cubic meters (DSCM) of air emitted in that time.

DATA SOURCE: Referenced E-Three, Inc. Report

Processing was done under 12 sets of conditions, each involving 3 to 4 drums of dredged material. Composite samples were taken for each set of process conditions as follows: For the solid samples (1S, 2S, 3S, 4S, 7S, 8S on Figure 10), three grab samples were taken from each of the drums associated with the process conditions at that point and composited to form one representative sample. Feed samples were taken from the top, middle, and bottom of each drum, while samples of treated sediment and residuals were taken at approximately the beginning, middle, and end of each run. Each composite was submitted for analysis of solids, total organic carbon (TOC), mercury, chromium, lead, oil and grease, PCBs, and 17 individual polycyclic aromatic hydrocarbons (PAHs). Additionally liquid samples were taken at the two points where liquid is removed from the process, namely: a removal point just past the processor inlet (9L on Figure 10) and a removal point just prior to the processor outlet (10L on Figure 10). In each case, the removed liquid was condensed, collected in a receiver, and the receiver contents for the entire run were well mixed prior to sampling. Liquid samples were analyzed for the same chemical parameters as were the solid samples.

Corps of Engineers air sampling was contracted to E-Three, Inc., and the resulting analyses were performed by Battelle Laboratories. No air sampling was performed directly by the Corps of Engineers.

TABLE 13  
ANALYTICAL PARAMETERS FOR SEDIMENT SAMPLES

Analysis	ARCS Required Detection Limit ug/g	Method	Battelle Instrument Detection Limit ug/g	Volume Required ml	Container
PAHs-16 Compounds by GC/MS	0.2	NOAA 1985	0.02	100	4 oz
PCBs (total Aroclors)	0.02	NOAA 1985	0.02	100	glass
Cr	2	PNL-SP-19B	1	50	Spex Jar or 32 oz plastic
Cu	2	PNL-SP-19B	1		
Hg	0.1	MSL-M-11	0.02		
Pb	2	PNL-SP-19B	1		
Total Organic Carbon	300	EPA 9060	0.10% (100 ug/g)	50	4 oz plastic
Total Solids	1000	EPA 160.3	0.10% (100 ug/g)	50	
Total Volatile Solids	1000	EPA 160.4	0.10% (100 ug/g)	50	
16 Fractions Grain Size	N/A	PSEP 1986	1.00%	100	8 oz plastic
Oil and Grease (Solvent Extractables)	N/A	EPA-LLRS- GROSSE	N/A	50	4 oz glass

References:

NOAA 1985 - NOAA 1985, Nation Oceanic and Atmospheric Administration, National Status and Trends Program, Standard Analytical Procedures.

- . PAHs: GC/MS, using selective Ions mode (S.I.M)
- . PCBs: GC/ECD using capillary columns

PNL-SP-19B - Energy Dispersive X-Ray Fluorescence Spectrometry (SOP # from Battelle Labs)

MSL-M-11 - Cold Vapor Atomic Absorption. (SOP # from Battelle Labs)

EPA 9060, TOC - U.S. Environmental Protection Agency (EPA). 1986. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. SW-846. U.S. Document No. 955-001-00000, USEPA, Washington, D.C.

EPA 160 - U.S. Environmental Protection Agency (EPA). 1983. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March, 1983, Method 413.2.

PSEP 1986 - Puget Sound Estuary Protocols.

ASTM-D422 - American Society of Testing Materials (ASTM). 1972. Standard Method for Particle-Size Analysis of Soil D-422. ASTM, Philadelphia, Pennsylvania.

EPA-LLRS-GROSSE - Procedure supplied by Great Lakes Large Lakes Lab, "Analysis of Solvent - Extractable Residue from Whole Sediment."

TABLE 14  
ANALYTICAL PARAMETERS FOR WATER SAMPLES

Analysis	ARCS Required Detection Limit ug/g	Method	Battelle Instrument Detection Limit ug/l	Volume Required ml	Container
PAHs-16 Compounds by GC/MS	2	NOAA 1985	0.02	1000	1000 ml glass
PCBs (total Aroclors)	0.01	NOAA 1985	0.01	1000	1000 ml glass
Cr	1	PNL-SP-24	1	500	500 ml Teflon
Cu	1	PNL-SP-24	1		
Hg	0.1	MSL-M-24	0.005		
Pb	1	PNL-SP-24	1		
Total Organic Carbon	1000	EPA 415.2	1000	200	60 ml glass
Total Solids	1000	EPA 160.3	1000	200	1000 ml
Total Suspended Solids	1000	EPA 160.1	1000	200	poly
pH	Full Range				200 ml plastic
Sp. Conductance	Full Range				
Oil & Grease	N/A	5520B	1.1 mg/l	1000	1000 ml glass

Additional References:

PNL-SP-24 - Metals and trace elements in water by Inductively Coupled -  
Mass Spectrometry (ICP/MS)

MSL-M-27 - Total mercury in water by CVAA

EPA 415.1 - Total Organic Carbon in water.

Method 5520B - "Standard Methods for the Examination of Water and  
Wastewater," 18th Edition, AWWA/WPCF.

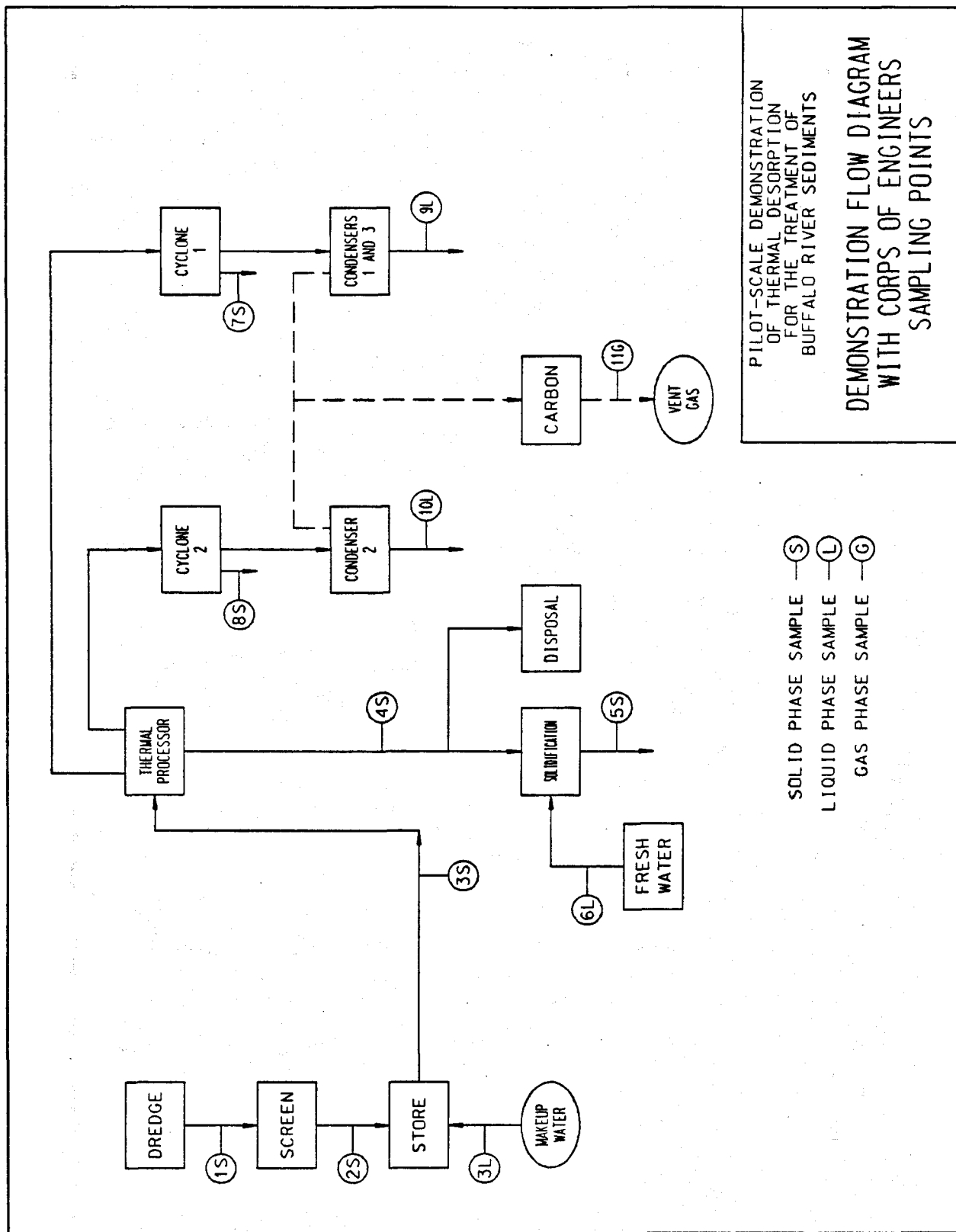


FIGURE 10



#### 2.12.3.2 Analytical Protocol--

Samples were analyzed using the analytical methods referenced in Tables 13 and 14. These methods were applied within the framework of a Quality Assurance Project Plan (QAPP) prepared by the U.S. Army Corps of Engineers Waterways Experiment Station. In general, this QAPP provided for replication of 5 percent of all field samples, performance of matrix spikes/matrix spike duplicates, and processing of all blanks associated with standard analytical lab practice. Both the analytical methodology and the quality assurance procedures generally proceeded per plan as determined by the Waterways Experiment Station and the Buffalo District Corps of Engineers. Most of the detection limits for analytes of concern were at or below the target limits given in Tables 13 and 14. Exceptions were detection limits for PAHs and PCBs. Detection limits for PAHs were 0.06 ug/g for sediment (versus the target 0.02 ug/g) and 0.04 ug/l for water (versus the target 0.02 ug/l). Detection limits for PCBs met the target values for sediment, but were 0.05 to 0.5 ug/l for water (versus the target 0.01 ug/l). Detection limit problems were caused by matrix effects that necessitated dilution of the samples in question.

Complete results of these analyses and the associated quality assurance testing are available in "Report of Chemical Analyses: Volumes 1-3," prepared for the USEPA Great Lakes National Program Office by Battelle Marine Research Laboratory. Summaries of these data, and an assessment of process performance based on these results, appear in subsequent sections of this report.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 CORPS OF ENGINEERS RESULTS

Reduced analytical data for the Buffalo Thermal Desorption Pilot Demonstration appear in subsequent sections of this report. Data is provided separately for each constituent of concern. Based upon presented results, an assessment is made regarding process efficiency for removing each chemical contaminant.

##### 3.1.1 Overall Mass Balance

An overall mass balance was performed on solids and liquids for this process. The intent was to measure the percent of feed material that could be accounted for after processing (i.e., percent closure). Results of this mass balance were reported by RETEC, and appear in Table 15. Closures of 100 percent  $\pm$  15 percent are desirable. For closures in this range, stream weights can be used with reasonable confidence to track individual compounds through the system and thus assess treatment effectiveness.

Based on acceptable closure constraints, all but Run B3 are suitable runs for determining process performance. Three additional runs were eliminated from consideration for other

TABLE 15  
SOLIDS/LIQUIDS MASS BALANCE

Run No.	Feed Material (lbs)	Treated(b) Material (lbs)	Total Condensate (lbs)	Percent Closure
A1	1896	970	765	93
A2	2010	1055	890	97
A3	2150	1235	688	89
B1	1355	845	620	108
B2	2175	1850	445	106
a B3	2180	585	465	48
C1	1640	880	840	105
C2	1905	955	880	96
C3	1940	950	850	93
D1	1515	700	950	109
D2	1150	480	550	90
D3	1630	955	790	107

- a. Closure is unacceptable for mass balance calculations  
b. This weight includes the weight of the solids which exited the cyclones.

DATA SOURCE: Referenced RETEC, Inc. Report

reasons. Run A1 was considered a startup run, so only a partial data set was taken. Due to budget constraints, Runs D2 and D3 were archived without performing analytical tests. Thus, all subsequent data is reported for Runs A2, A3, B1, B2, C1, C2, C3, and D1.

### 3.1.2 Solids Content

Total solids data is given in Tables 16A and 16B and volatile solids data is presented in Table 17. Graphs of these parameters versus final solids temperatures are given in Figures 11 and 12 (Error bars shown are for the standard error of the mean). From these data, it is clear that feed material containing 44-56 percent solids was successfully pumped and dried such that final solids content normally exceeded 95 percent. Maximum sediment temperatures employed to achieve these results varied from 300°F to 480°F and residence times varied from 30 to 90 minutes.

Because one project goal was to obtain information for design and operation of a full scale process, it was desired to determine whether removal efficiencies were a function of any of the process variables monitored for the study. To assess effects of process variables, correlation coefficients were determined relating removal to each measured process parameter. (The formula used to calculate the correlation coefficient is given in Appendix A).

TABLE 16A  
PERCENT TOTAL SOLIDS IN SEDIMENT

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	58.0	58.0	55.2	55.2	59.8	59.8	59.8	56.9
After Screening (2s)	63.0	60.2	63.2	62.0	63.8	63.9	64.8	58.2
Before Thermal Proc. (3s)	55.4	55.0	56.1	54.6	52.1	50.8	48.3	44.6
After Thermal Proc. (4s)	99.8	99.5	98.6	99.8	99.8	99.9	95.2	99.9
Solids from 1st Cyclone (7s)	95.3	97.5	99.1	87.1	83.0	95.3	85.1	94.2
Solids from 2nd Cyclone (8s)	77.1	99.6	99.5	99.5	98.4	N/A	99.6	99.7

TABLE 16B  
PERCENT TOTAL SOLIDS IN CONDENSATE

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	1.1	N/A	N/A	0.7	0.9	0.5	0.9	0.4
After 2nd Condenser (10L)	0.8	0.8	0.8	0.2	0.2	0.1	0.1	0.2

\* Process conditions same as for sediment

N/A = Not Analyzed

Highlighted values are average of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

TABLE 17  
PERCENT VOLATILE SOLIDS IN SEDIMENT

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	6.4	6.4	6.2	6.2	5.1	5.1	5.1	5.5
After Screening (2s)	6.0	6.0	5.4	5.4	4.9	5.3	5.5	5.4
Before Thermal Proc. (3s)	5.6	5.6	5.5	5.5	5.4	5.6	5.0	4.9
After Thermal Proc. (4s)	3.4	4.2	3.7	4.3	4.3	3.7	4.1	3.4
Solids from 1st cyclone (7s)	4.7	5.4	5.1	3.8	4.9	4.9	5.2	5.1
Solids from 2nd Cyclone (8s)	2.3	4.7	5.0	3.8	4.2	N/A	4.8	4.5

$$\% \text{ Volatile Solids} = \frac{(A - D)}{A} \times 100$$

WHERE: A = weight of residue after drying at 105°C for 1 hour  
D = weight of residue after dried residue has been ignited at 550°C for 1 hour

Highlighted values are the averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

N/A = Not Analyzed.

FIGURE 11  
% SOLIDS VERSUS EXIT TEMPERATURE OF SOLIDS  
(NUMBERS IN PARENTHESES ARE NUMBERS OF DATA POINTS  
FROM WHICH THE STANDARD ERRORS WERE CALCULATED)

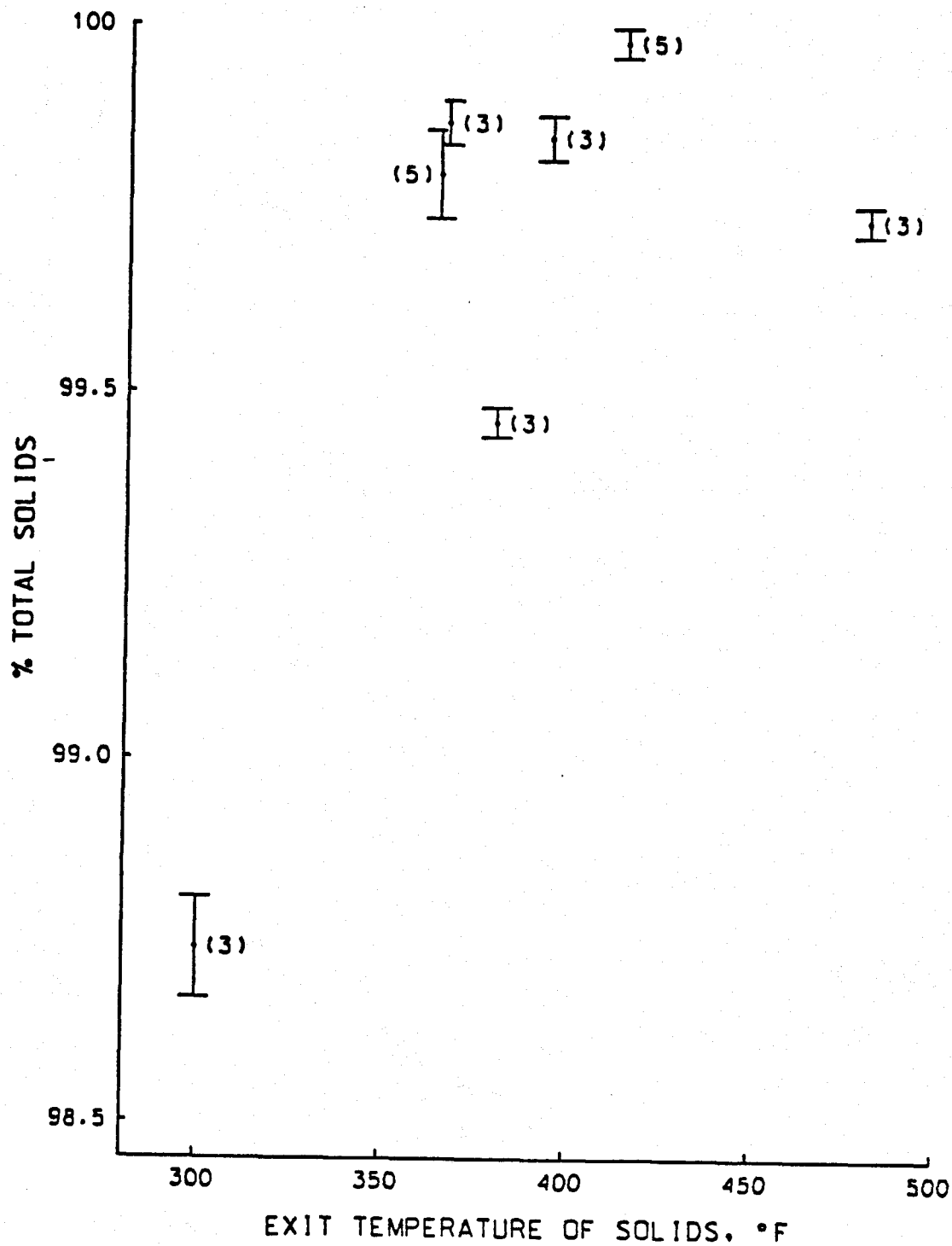
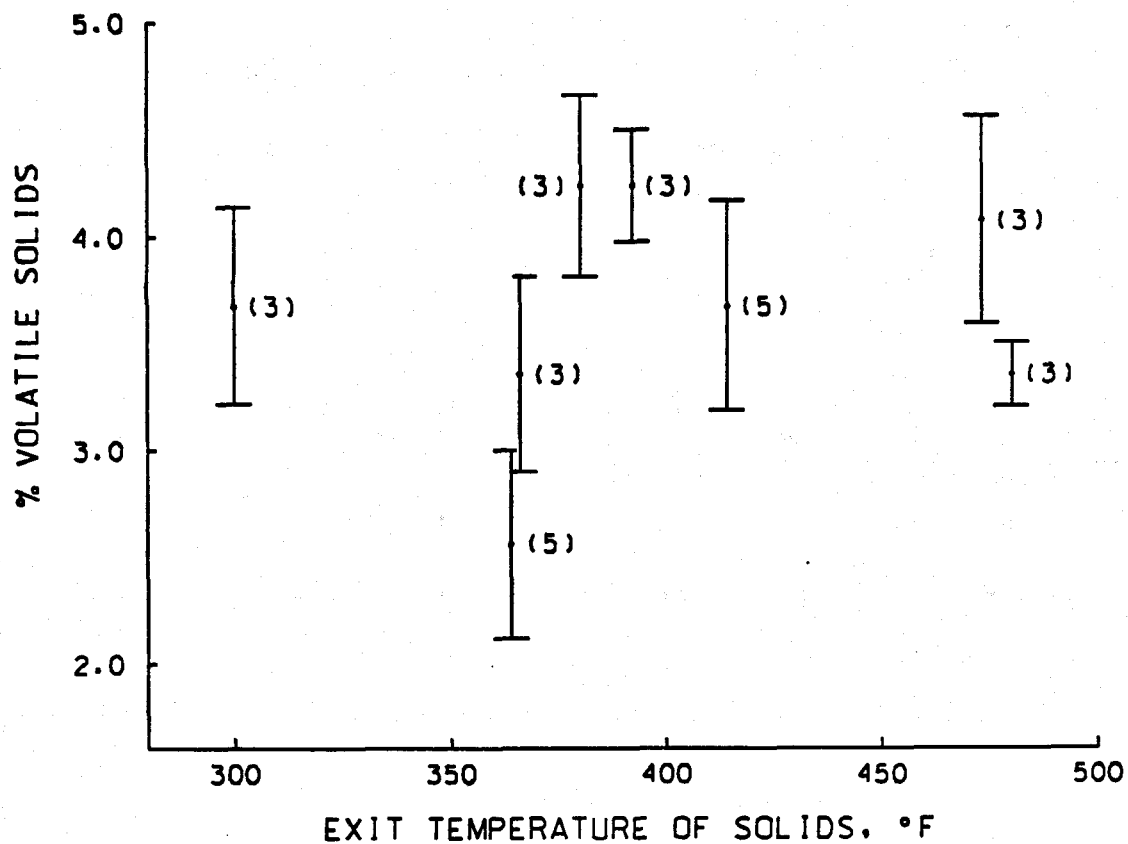


FIGURE 12  
% VOLATILE SOLIDS VERSUS EXIT TEMPERATURE OF SOLIDS  
(NUMBERS IN PARENTHESES ARE NUMBERS OF DATA POINTS  
FROM WHICH THE STANDARD ERRORS WERE CALCULATED)



A correlation coefficient of  $\pm 1.0$  means that process performance can be perfectly predicted if the value of the process variable is known. An r value of 0 means that process performance and the process variable are completely unrelated. (Correlation coefficients ranging from 0 to +1.0 apply to variables related by a straight line with a positive slope and correlation coefficients from 0 to -1.0 apply to variables that are related by a straight line with a negative slope.) Correlation coefficient values with an absolute value higher than 0.9 are usually considered as acceptable for prediction of process performance. Correlation coefficients relating process parameters with volatiles removal are given in Table 18.

TABLE 18  
CORRELATION COEFFICIENTS FOR VOLATILE SOLIDS REMOVAL

Parameter Correlated with Total Volatile Solids (TVS) Removal	Correlation Coefficient
Process Temperature	0.40
Residence Time	0.40
Initial Volatiles Solid Content	-0.34

Based on these results, it is clear that some other phenomenon besides initial liquids content and simple energy input is dictating extent of drying of sediment and therefore extent of contaminant removal. It is possible that some threshold energy input is required before complete removal of these materials is achieved, and that the threshold was not reached with the residence times and temperatures employed for this study. RETEC's thermal desorption process is capable of achieving maximum sediment temperatures of 700-800°F, at the expense of throughput rate. Should this process be tried again, it is suggested that trials at higher temperatures take place.

### 3.1.3 Metals

An overall material balance was performed on each run for each metal of concern. A sample calculation is provided for Run A3, for chromium, as follows:

#### (a) Adjustment of Measured Stream Weights to a Dry Weight Basis

Mass of Input:

$$(2150 \text{ lbs}) \times \frac{(0.550 \text{ lbs dry solids})}{\text{lb}} \times \frac{(0.454 \text{ kg})}{\text{lb}} = 537 \text{ kg dry solids}$$

Mass of Output/Solid Stream (Average percent solids in treated solids plus residue from cyclones):

$$(1235 \text{ lbs}) \times \frac{(0.995 \text{ lbs dry solids})}{\text{lbs}} \times \frac{(0.454 \text{ kg})}{\text{lb}} = 557 \text{ kg dry solids}$$

Mass of Solid Output in Liquid Stream:

$$(688 \text{ lbs liquid}) \times \frac{(0.008 \text{ lb solids})}{1 \text{ lb liquid}} \times \frac{(0.454 \text{ kg})}{1 \text{ lb}} = 2.5 \text{ kg dry solids}$$

(b) Material Balance on Chromium for Run A3

Input:

$$(537,000 \text{ g solids}) \times \frac{(68 \times 10^{-6} \text{ g Cr})}{\text{g solids}} = 36.5 \text{ g Cr}$$

Since it is not known what percent of total solids came from the cyclones (they were combined and then analyzed), the following is assumed for these calculations:

cyclone 1: 4%

cyclone 2: negligible

Overall concentration of processed solids is therefore:  
 $0.96(61) + 0.04(148) = 65 \text{ ug/g}$

Where 61 ug/g and 148 ug/g are analytical values

Cr in Treated Solids:

$$(557,000 \text{ g}) \times \frac{(65 \times 10^{-6} \text{ g Cr})}{\text{g}} = 36.2 \text{ g}$$

Cr in Condensate Solids:

$$(2,500 \text{ g}) \times \frac{(148 \times 10^{-6} \text{ g Cr})}{\text{g}} = 0.37 \text{ g (negligible)}$$

Cr in Condensate:

$$(688 \text{ lbs}) \times \frac{(0.454 \text{ kg})}{1 \text{ lb}} = 312 \text{ kg}$$

$$(312 \text{ kg}) \times \frac{(1 \text{ L})}{\text{kg}} \times \frac{(14,200 \times 10^{-6} \text{ g Cr})}{\text{L}} = 4.4 \text{ grams}$$

Percent Removed from Sediment:

$$\frac{(36.5 \text{ g} - 36.2 \text{ g})}{36.5 \text{ g}} \times 100 = 0.8\% \text{ removal}$$

Analytical results for metals are summarized in Tables 19A and 19B thru 22A and 22B. From these results, it is clear that removal levels are somewhat metal specific. Copper, lead, and chromium are discussed together because they behave similarly under conditions employed for this study. Mercury is discussed separately because its behavior is distinctly different from the other metals.

#### Copper, Lead, and Chromium

For copper, lead, and chromium, residue concentrations in ug/g did not change appreciably from those in the feed although



TABLE 19A  
LEAD IN SEDIMENT  
(ug/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	72.2	72.2	49.3	49.3	88.8	88.8	88.8	49.7
After Screening (2s)	52.4	52.4	58.2	58.2	62.3	62.3	62.3	62.3
Before Thermal Proc. (3s)	53.7	52.4	64.3	61.7	68.4	71.9	56.3	73.5
After Thermal Proc. (4s)	52.1	52.7	65.7	67.0	70.6	67.2	58.3	64.7
Solids from 1st Cyclone (7s)	58.9	56.7	65.1	67.0	83.8	70.7	70.4	63.6
Solids from 2nd Cyclone (8s)	48.8	48.0	58.4	64.5	59.7	N/A	61.7	71.8

TABLE 19B  
LEAD IN CONDENSATE  
(Filtered)  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
After 2nd Condenser (10L)	2.97	1.19	3.56	2.37	1.78	14.0	17.8	8.90

N/A Not Analyzed

\* Process conditions same as for sediment

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

TABLE 20A  
CHROMIUM IN SEDIMENT  
(ug/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	73	73	63	63	80	80	80	74
After Screening (2s)	60	60	51	51	71	71	71	60
Before Thermal Proc. (3s)	47	68	47	58	73	59	69	55
After Thermal Proc. (4s)	70	61	56	44	67	58	62	62
Solids from 1st Cyclone (7s)	137	148	129	106	136	129	140	112
Solids from 2nd Cyclone (8s)	84	88	121	77	129	N/A	140	118

TABLE 20B  
CHROMIUM IN CONDENSATE  
(Filtered)  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
After 2nd Condenser (10L)	4.2	2.9	2.52	1.76	2.78	1.80	1.01	1.64

N/A Not Analyzed

\* Process conditions same as for sediment

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

TABLE 21A  
COPPER IN SEDIMENT  
(ug/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	46.2	46.2	41.0	41.0	56.8	56.8	56.8	37.7
After Screening (2s)	45.8	45.8	43.0	43.0	47.8	47.8	47.8	45.0
Before Thermal Proc. (3s)	41.1	40.6	37.6	43.0	45.3	48.2	47.2	44.8
After Thermal Proc. (4s)	38.2	41.3	45.7	45.6	58.0	48.8	45.4	47.6
Solids from 1st Cyclone (7s)	51.3	47.1	48.2	46.8	54.8	56.1	62.4	51.3
Solids from 2nd Cyclone (8s)	42.3	43.1	46.2	47.1	62.1	N/A	52.5	53.7

TABLE 21B  
COPPER IN CONDENSATE  
(Filtered)  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
After 2nd Condenser (10L)	830	410	340	450	720	120	70	755

N/A Not Analyzed

\* Process conditions same as for sediment

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

TABLE 22A  
MERCURY IN SEDIMENT  
(ug/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	0.18	0.18	0.17	0.17	0.34	0.34	0.34	0.11
After Screening (2s)	0.19	0.19	0.21	0.21	0.20	0.20	0.20	0.21
Before Thermal Proc. (3s)	0.17	0.18	0.20	0.20	0.20	0.21	0.18	0.19
After Thermal Proc. (4s)	0.00	0.05	0.11	0.00	0.07	0.05	0.15	0.01
Solids from 1st Cyclone (7s)	0.15	0.12	0.13	0.10	0.32	0.22	0.49	0.21
Solids from 2nd Cyclone (8s)	0.02	0.04	0.12	0.02	0.30	N/A	0.06	0.03

TABLE 22B  
MERCURY IN CONDENSATE  
(Filtered)  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	(44.07)	3.26	N/A	5.28	9.30	2.16	0.01	3.20
After 2nd Condenser (10L)	2.50	4.00	0.95	1.44	1.34	3.66	1.81	3.37

N/A Not Analyzed

\* Process conditions same as for sediment

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

concentrations of lead and chromium decrease significantly from dredging to screening. Although not determined experimentally, it is postulated that some of the metals precipitated and adhered to the side-walls of the storage drum. During treatment, all three metals tend to remain with the treated residue, and are thus found either in the bulk of the treated residue or in the treated solids captured by the cyclones. For copper and lead, concentration of the cyclone solids is approximately the same as concentration of the bulk residue. For chromium, the concentration is greater in the cyclone solids than in the bulk residue, suggesting that chromium tends to associate with the finer particulates. Concentrations of Cu, Pb, and Cr in condensate are negligible. In summary, copper, chromium and lead tend to remain with the solids and are potential candidates for stabilization.

One of the goals for this study was to assess contaminant losses during processing. For this reason, a mass balance was performed on each metal for each process step to determine fate of the metals that were removed by processing. That is, if the constituent was desorbed from the residue, where did it go and how must it be captured to prevent further environmental contamination? A sample mass balance calculation is given in Appendix A. Table 23 summarizes the mass balance results for Cu, Pb, Cr, and Hg.

From these results it is clear that the majority of these metals remain with the residue. As can be seen from these results, mass balance closures are variable. Sometimes all material is not accounted for, while at other times the output of key constituents of concern appears to exceed the input. There are several reasons why this is true.

- (1) Most constituents of concern for this project were present in extremely small quantities. For example, the total quantity of chromium being tracked through the process for an entire run was in the range of 50 grams. For such small contaminant quantities, the sum of the extremely small errors inherent in the analytical procedures comprises a significant percentage of the total amount present.

- (2) Individual weights of the solid streams were not taken, rather all solids were combined and a single weight was obtained. Since compositions of these individual streams were not the same, the act of combining these streams resulted in an approximate rather than an exact material balance.

- (3) Air emissions analysis did not include analysis for metals, thus metal losses into the atmosphere or within the carbon adsorber were not quantified. Indeed, the carbon adsorber is a sink that was not accounted for in the sampling and analysis program.

TABLE 23A  
SUMMARY OF CALCULATIONS  
CHROMIUM

<--- GRAMS OUT --->				
BIN	GRAMS IN	RESIDUE	CONDENSATE	PERCENT REMOVED
A2	23.7	34.8	5.2	0
A3	36.5	36.2	4.4	0.8
B1	16.2	22.3	0.5	0
B2	31.3	39.4	0.0	0
C1	28.3	27.9	0.3	1.4
C2	25.9	26.4	0.2	0
C3	29.3	26.7	0.4	8.8
D1	16.9	20.3	0.3	<u>0</u>
AVG				1.4
<u>No significant change</u>				

TABLE 23B  
SUMMARY OF CALCULATIONS  
COPPER

<--- GRAMS OUT --->				
BIN	GRAMS IN	RESIDUE	CONDENSATE	PERCENT REMOVED
A2	20.7	18.4	10.3	11.1
A3	21.8	23.0	2.2	0
B1	13.0	17.3	0.2	0
B2	23.1	38.5	0.0	0
C1	17.5	23.0	1.4	0
C2	21.1	21.2	0.9	0
C3	20.0	18.9	0.8	5.5
D1	13.8	15.2	0.3	<u>0</u>
AVG				2.0
<u>No significant change</u>				

TABLE 23C  
SUMMARY OF CALCULATIONS  
LEAD

BIN	GRAMS IN	<--- GRAMS OUT --->		PERCENT REMOVED FROM SEDIMENT
		RESIDUE	CONDENSATE	
A2	27.1	25.0	5.1	7.7
A3	28.1	29.5	2.2	0
B1	22.2	24.7	0.0	0
B2	33.3	56.1	0.0	0
C1	26.5	28.3	0.3	0
C2	31.6	29.1	0.1	7.9
C3	23.9	24.2	0.1	0
D1	22.6	20.5	0.2	<u>9.3</u>
AVG				3.1

No significant change

TABLE 23D  
SUMMARY OF CALCULATIONS  
MERCURY

BIN	GRAMS IN	<--- GRAMS OUT --->		PERCENT REMOVED FROM SEDIMENT
		RESIDUE	CONDENSATE	
A2	.086	.005	0.03	94.1
A3	.094	.028	0	70.2
B1	.070	.042	0	40.0
B2	.107	0	0	100
C1	.078	.030	0	61.5
C2	.094	.010	0	89.4
C3	.074	.067	0	9.5
D1	.057	.007	0	<u>87.7</u>
AVG				69.2

(4) The process equipment used for this demonstration was, of necessity, portable equipment that is often transported, disassembled, and reassembled. For this reason, areas near seals were not as air-tight as they tend to be when assembly and disassembly are infrequent. Some fine particulate losses were observed at the seal where the conveyor dome bolts to the conveyor body. Since the constituents of concern tended to be either volatile or associated with fine particulates, these small losses could affect the material balance. It must be emphasized that losses of this nature did not result in measurable discharges of pollutants to the environment. The contaminant input to the process equipment was so small that it was barely measurable, thus losses that affected material balance closures were trace amounts indeed.

For future work, it is recommended that air emissions losses for metals be performed and that process streams be weighed separately before they are combined for disposal. This should help improve mass balance closure somewhat and should permit calculation of air emissions. As for the other sources of error, problem (4) will be greatly minimized for full scale, permanently installed equipment. The impact of problem (1) can be minimized for highly contaminated sediments, but cannot be corrected by process methodology.

### Mercury

Following processing, the bulk of the treated residue was free of mercury as were the solids from the second cyclone and the condensate. Mercury content of solids from the first cyclone was approximately the same as the untreated solids. Based on mass balances, however, an average of 79 percent of the mercury in the feed was unaccounted for after processing. Reasons why the fate of mercury is largely unknown are the same as reasons why closure was not obtained for chromium, copper, and lead. As for these other metals, closure can be somewhat improved by weighing each process stream and by quantifying air emissions. Air emissions monitoring will be even more important for mercury, however, because it is a liquid at room temperature and has a relatively high vapor pressure. Again, equipment leaks and errors due to low initial concentrations are inherent in the process and cannot be eliminated in a pilot study of this nature.

#### 3.1.4 Polycyclic Aromatic Hydrocarbons (PAHs)--

Concentrations of 17 individual PAHs were measured both before and after processing, and an attempt was made to assess both removal efficiency and ultimate fate of the removed materials. (Although not confirmed experimentally, it is likely that PAH concentrations increased between dredging and screening due to concentration effects of liquid evaporation.) Where a concentration was reported as "less than detection limit," the detection limit itself was the concentration used in the summation. This approach was used to assure that sediment disposal questions addressed for process scaleup would provide maximum environmental protection. To facilitate this analysis, individual PAH results were summed in two groups.



. Low molecular weight PAHs, i.e., those containing  $\leq 3$  aromatic rings (naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene).

. High molecular weight PAHs, i.e., those containing  $> 3$  aromatic rings (fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)-pyrene, dibenzo(a,h)-anthracene, and benzo(g,h,i)-perylene).

Individual compounds were grouped this way primarily because many PAHs with  $> 3$  aromatic rings are either known or suspected carcinogens, thus their fate and removal efficiencies are of particular interest.

PAH removal efficiencies are discussed by group, as follows:

#### Low Molecular Weight PAHs (LMW PAHs)

Results of low molecular weight PAH removal are given in Tables 24A and 24B. A graph of LMW PAH concentration versus final solids temperature is given in Figure 13 (Error bars shown are for standard error of the mean). From these data it is determined that a range of 45 to 90 percent of the PAHs with  $\leq 3$  aromatic rings were removed from the feed material as a result of thermal desorption. Correlation of removal with process temperatures was not significant, as the correlation coefficient relating the two variables was 0.00. It is not known whether a larger energy input would remove the remaining LMW PAHs from the sediment. Future work involving low temperature thermal desorption should include some higher temperatures in order to assess the effect of this process parameter.

As for fate of the removed PAHs, concentration of LMW PAHs in the solids captured by the cyclones is approximately that found in the unprocessed feed. Negligible amounts of LMW PAHs are found in the condensate (average = 0.14 ug/g) and in the air emissions (range is 41.1-213.6 ug/DSCM). Net result is that approximately 74 percent of the PAHs originally present in the feed are not accounted for by mass balance (Table 26A). Closure was not obtained for LMW PAHs. The first three reasons for low percent closure given in section 3.1.3 apply here as well. Additionally, it is speculated that much of the unaccounted-for PAHs sorbed to the carbon. The LMW PAH compounds have an activated carbon: water partition coefficient ( $P_{ac}$ ) of  $10E4.7$  to  $10E6.8$  (Verscheuren, 1983). This range of  $P_{ac}$  values suggests a strong tendency for carbon to adsorb airborne PAHs from the exhaust air. For any future work of this nature, it would be well to attempt identifying and quantifying the chemicals adsorbed by the carbon system. Desorption of these materials and their subsequent quantification may be impractical, but if investigation of the problem results in disclosure of a practical methodology, the resulting data will be valuable.

TABLE 24A  
LOW MOLECULAR WEIGHT ( $\leq 3$  RING)  
PAHs IN SEDIMENT  
(ng/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	791	791	926	926	1390	1390	1390	937
After Screening (2s)	1538	990	950	1170	1577	1221	1221	9994
Before Thermal Proc. (3s)	1124	1177	842	1367	1428	1164	1039	2018
After Thermal Proc. (4s)	122	210	349	107	211	260	425	102
Solids from 1st Cyclone (7s)	1947	838	1417	1625	N/A	822	4493	2689
Solids from 2nd Cyclone (8s)	1105	327	1430	613	1663	N/A	369	576

TABLE 24B  
LOW MOLECULAR WEIGHT ( $\leq 3$  RINGS)  
PAHs IN CONDENSATE  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	215	361	N/A	20	149	205	45	65
After 2nd Condenser (10L)	230	147	209	68	N/A	55	159	35

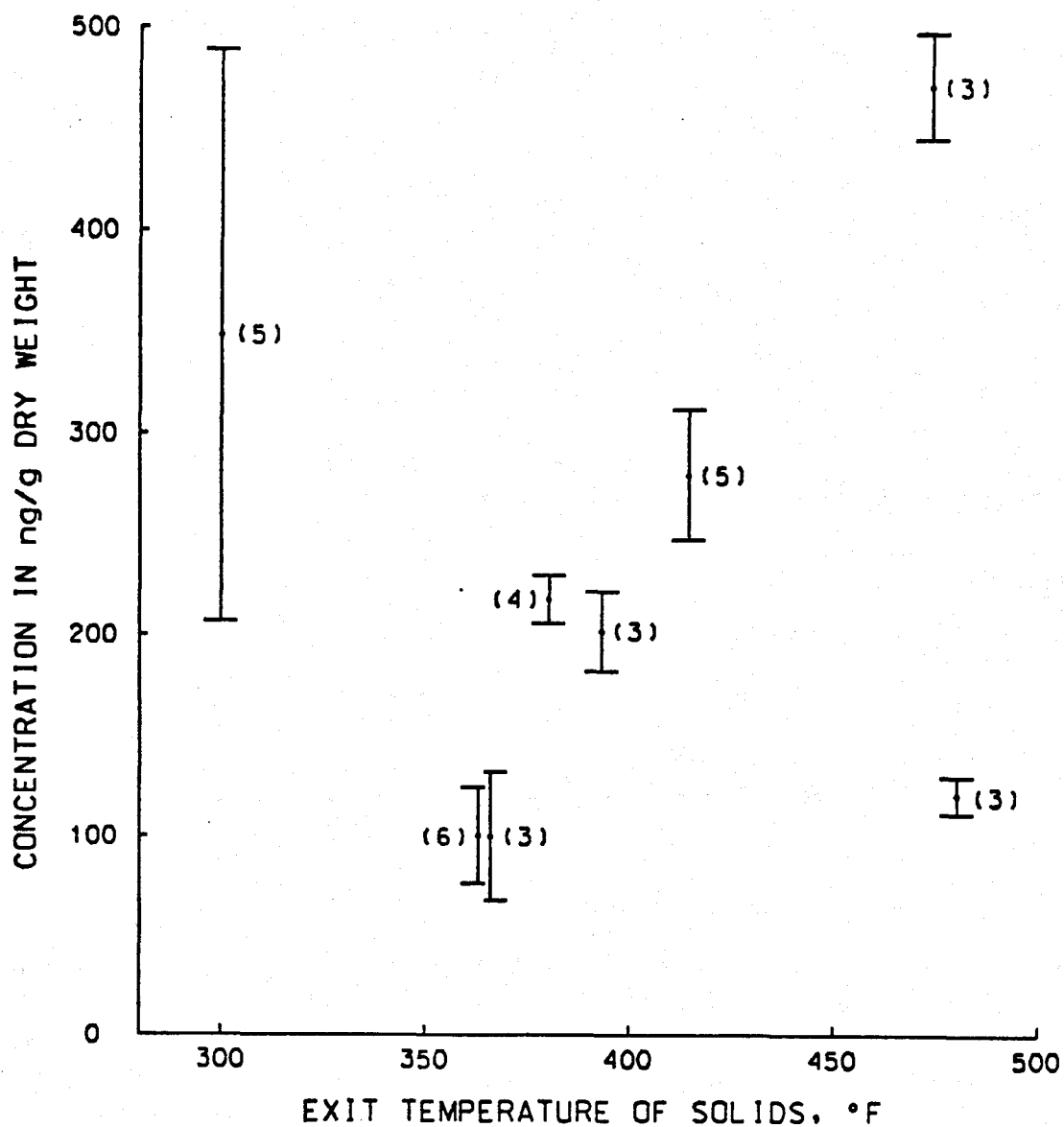
\* Process conditions same as for sediment

N/A Not Analyzed

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

FIGURE 13  
CONCENTRATION OF LOW MOLECULAR WEIGHT PAH<sup>S</sup> IN SEDIMENT  
VERSUS EXIT TEMPERATURE OF SOLIDS  
(NUMBERS IN PARENTHESES ARE NUMBERS OF DATA POINTS  
FROM WHICH THE STANDARD ERRORS WERE CALCULATED)



### High Molecular Weight PAHs (HMW PAHs)

High molecular weight PAH removal results are given in Tables 25A and 25B. A graph of HMW PAH concentration versus final solids temperature is given in Figure 14 (Error bars shown are for standard error of the mean). Based on these results, it is concluded that 72 percent of all PAHs with carcinogenic potential have been removed by this process (Table 26B). As with other contaminants of concern, HMW PAH removal does not correlate well with maximum sediment temperature. The correlation coefficient relating these variables was 0.00. As was stated for LMW PAH removal, effect of higher temperature on removal should be assessed for future work.

As for the fate of removed HMW PAHs, concentration of these materials in the first cyclone is approximately that found in the unprocessed feed. Concentration of HMW PAHs in the second cyclone is between that in the unprocessed feed and that in the bulk of the treated residue. Negligible amounts of HMW PAHs are found in the condensate (average = 0.15 ug/g) and in the air emissions (range is  $8.9\text{E-}07$  to  $2.3\text{E-}05$  ug/DSCM). Net result is that 71.6 percent of the HMW PAHs in the feed are unaccounted-for by the overall mass balance (Table 25B). The same reasons for low percent closure that applied to LMW PAHs apply here, except that tendency to sorb to carbon is stronger for the HMW PAHs ( $P_{ac}$  for HMW PAHs is  $10\text{E}6.5$  to  $10\text{E}7.0$ ).

### Total PAHs

Total PAH removal results are given in Tables 27A and 27B. Based on these results, from 43-94 percent of the total PAH contaminants were removed by the thermal desorption process.

Fate of these materials has been discussed previously under sections pertaining to Low Molecular Weight PAHs and High Molecular Weight PAHs and is not repeated here. Tables showing removals of specific PAHs are given in Appendix A.

#### 3.1.5 Solvent Extractables (SE)

Results of solvent extractables removal, as measured by standard oil and grease analysis, are presented in Tables 28A and 28B. A graph of solvent extractable concentration versus solids temperature is given in Figure 15. Error bars shown are for standard error of the mean. From these data it is determined that 17-86 percent of the oil and grease was removed from the residue, with an average removal of 68 percent. SE removal did not correlate well with maximum sediment temperature, as the correlation coefficient relating temperature and removal was -0.45. It is not known whether use of higher temperatures could improve removal. While low correlation of removal with temperature was true for temperatures  $\leq 480^{\circ}\text{F}$ , it may be true that some specific additional energy input is required before removal of SE can occur.

TABLE 25A  
HIGH MOLECULAR WEIGHT (>3 RINGS)  
PAHs IN SEDIMENT  
(ng/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	5424	5424	6604	6604	7652	7652	7652	6479
After Screening (2s)	7860	6325	5787	7187	7519	6600	6804	41,871
Before Thermal Proc. (3s)	6990	5529	5621	6502	7198	5985	5952	9535
After Thermal Proc. (4s)	119	1425	1664	61	1608	1865	3549	450
Solids from 1st Cyclone (7s)	4354	8811	11,679	7610	N/A	8019	21,178	25,499
Solids from 2nd Cyclone (8s)	1588	3938	2300	4349	6943	N/A	3043	6744

TABLE 25B  
HIGH MOLECULAR WEIGHT (>3 RINGS)  
PAHs IN CONDENSATE  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	156	235	N/A	7	129	218	82	111
After 2nd Condenser (10L)	268	190	245	59	N/A	75	220	42

N/A Not Analyzed

\* Process conditions same as for sediment

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

TABLE 26A  
SUMMARY OF CALCULATIONS  
LOW MOLECULAR WEIGHT PAHs

<--- MILLIGRAMS OUT --->				
BIN	MGMS IN	RESIDUE	CONDENSATE	PERCENT REMOVED
A2	568	93	0.08	83.7
A3	632	82	0.11	87.1
B1	290	145	0.06	50.0
B2	737	141	0.00	80.9
C1	554	107	0.06	80.6
C2	511	122	0.08	76.1
C3	442	242	0.02	45.3
D1	620	65.1	0.02	<u>89.5</u>
AVG				74.2

TABLE 26B  
SUMMARY OF CALCULATIONS  
HIGH MOLECULAR WEIGHT PAHs

<--- MILLIGRAMS OUT --->				
BIN	MGMS IN	RESIDUE	CONDENSATE	PERCENT REMOVED
A2	3530	138	0.06	96.1
A3	2969	958	0.07	67.7
B1	1939	782	0.07	59.7
B2	3504	305	0.00	91.3
C1	2793	725	0.05	74.1
C2	2627	914	0.09	65.2
C3	2487	1436	0.03	42.2
D1	2927	734	0.03	<u>74.9</u>
AVG				71.4

FIGURE 14  
CONCENTRATION OF HIGH MOLECULAR WEIGHT PAH<sup>S</sup> IN SEDIMENT  
VERSUS EXIT TEMPERATURE OF SOLIDS  
(NUMBERS IN PARENTHESES ARE NUMBERS OF DATA POINTS  
FROM WHICH THE STANDARD ERRORS WERE CALCULATED)

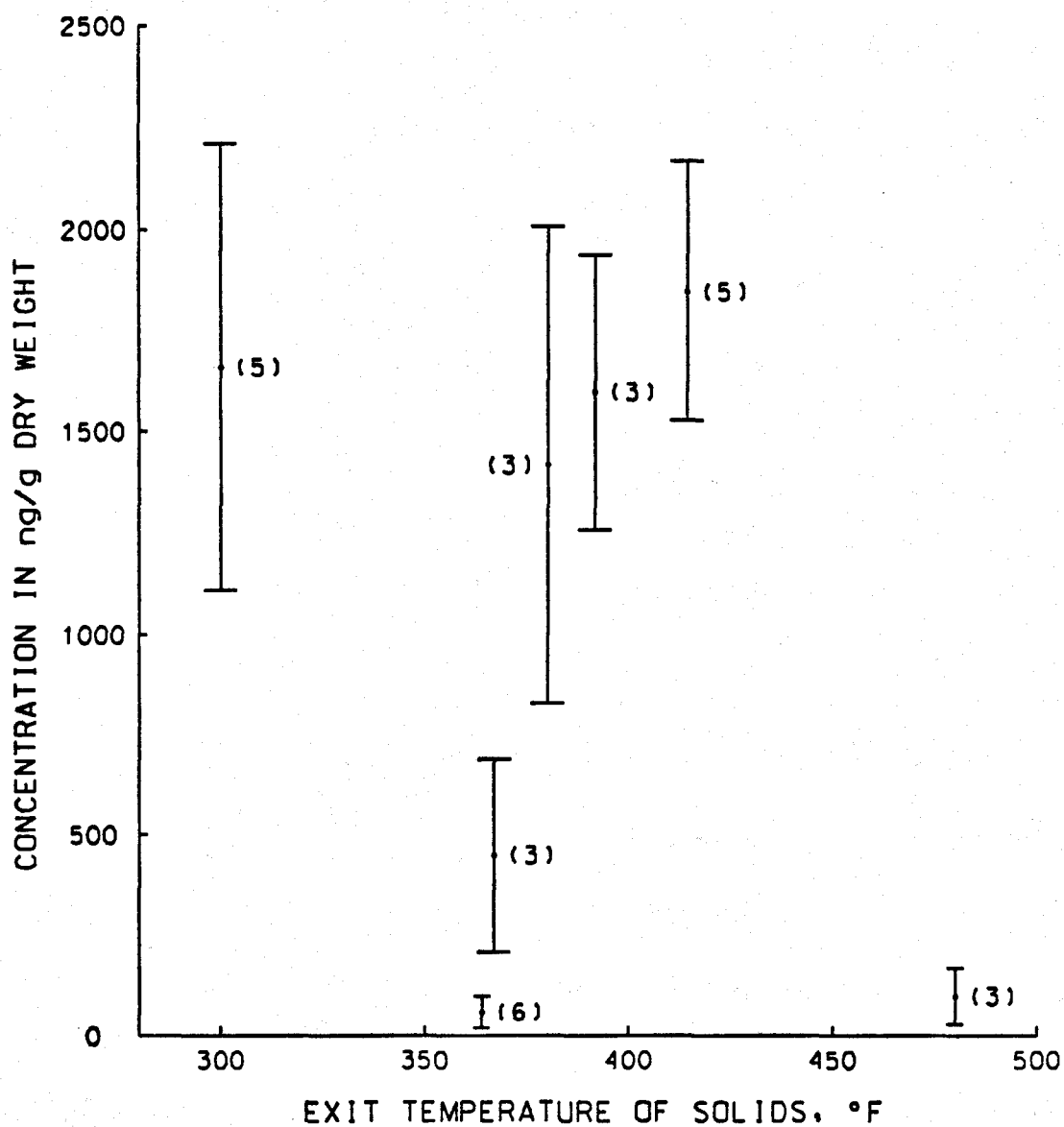


TABLE 27A  
TOTAL PAHs IN SEDIMENT  
(ng/g dry weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	6215	6215	7530	7530	9042	9042	9042	7416
After Screening (2s)	9398	7315	6737	8357	9096	7821	8025	51865
Before Thermal Proc. (3s)	8114	6706	6463	7869	8626	7149	6991	11553
After Thermal Proc. (4s)	241	1635	2013	168	1819	2125	3974	552
Solids from 1st Cyclone (7s)	6301	9649	13096	9235	N/A	8841	25671	5238
Solids from 2nd Cyclone (8s)	2693	4265	3730	4962	8606	N/A	3412	7320

TABLE 27B  
TOTAL PAHs IN CONDENSATE  
(ug/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
Liq. from 1st Condenser (9L)	371	596	N/A	20	278	423	127	176
Liq. from 2nd Condenser (10L)	498	337	454	127	N/A	130	379	77

N/A - Not Analyzed

\* Process conditions same as for sediment

Highlighted values are averages of multiple (3-5) measurements.

DATA SOURCE: Referenced Battelle Report



TABLE 28A  
SOLVENT EXTRACTABLES IN SEDIMENT  
(ug/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	1415	1415	1619	1619	2261	2261	2261	1707
After Screening (2s)	1820	1766	2006	2082	3995	1761	2399	2416
Before Thermal Proc. (3s)	2282	2250	2056	1754	2109	2011	4254	2391
After Thermal Proc. (4s)	234	550	474	220	562	510	3618	646
Solids from 1st Cyclone (7s)	2391	2443	3770	1231	N/A	2416	4389	5368
Solids from 2nd Cyclone (8s)	696	1901	1899	449	2539	N/A	646	965

TABLE 28B  
SOLVENT EXTRACTABLES IN CONDENSATE  
(mg/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	284	112	N/A	62	90	82	113	57
After 2nd Condenser (10L)	110	214	29	70	N/A	30	16	71

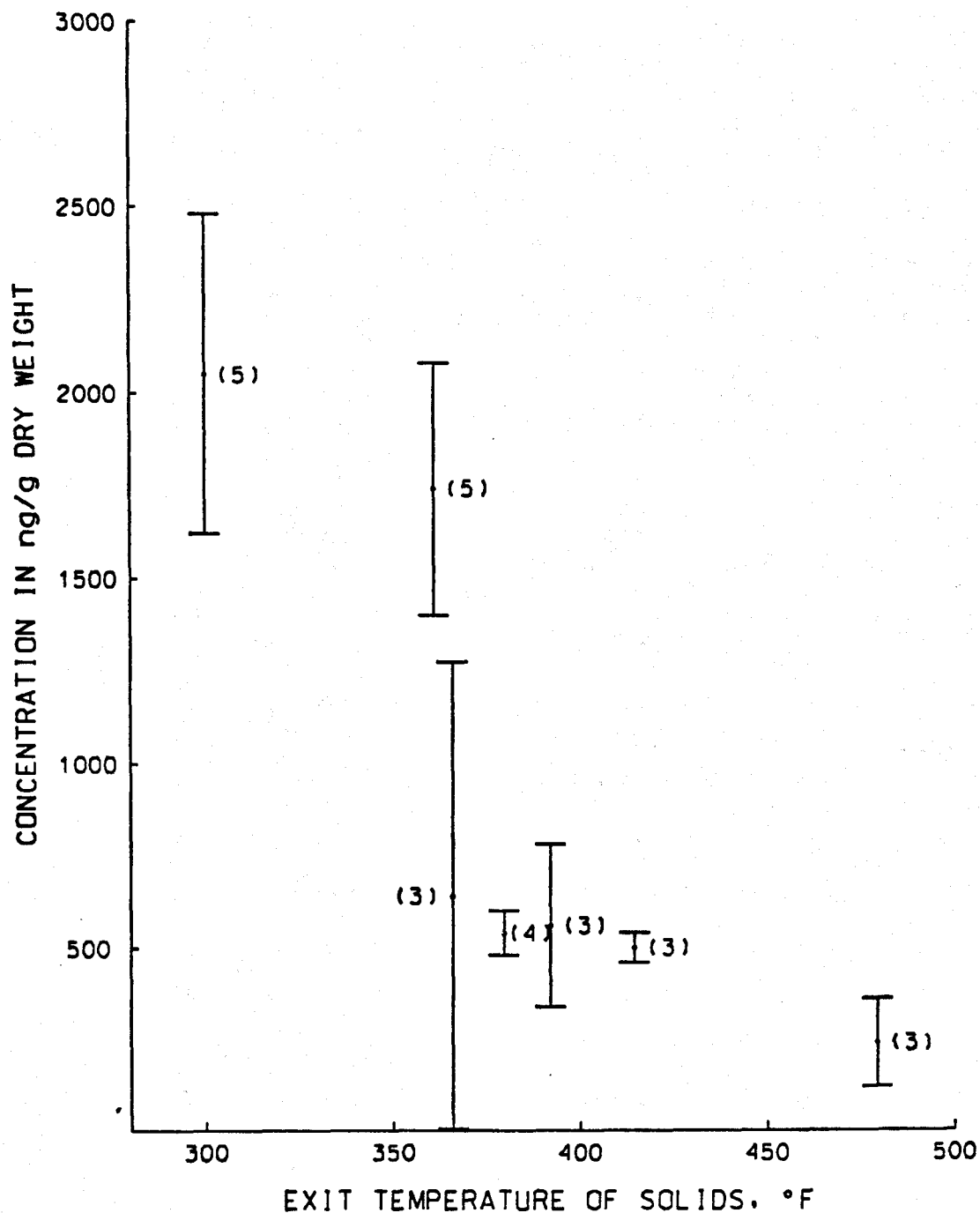
\* Process conditions same as for sediment

N/A - Not Analyzed

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

FIGURE 15  
CONCENTRATION OF SOLVENT EXTRACTABLES IN SEDIMENT  
VERSUS EXIT TEMPERATURE OF SOLIDS  
(NUMBERS IN PARENTHESES ARE NUMBERS OF DATA POINTS  
FROM WHICH THE STANDARD ERRORS WERE CALCULATED)



As for the fate of oil and grease, it tends to be concentrated in the fines captured by the first cyclone. Still, 68 percent of the removed oil and grease is unaccounted for (Table 29). It is known that negligible amounts appear in the condensate and in the fines from the second cyclone, however, amounts sorbed on carbon and amounts in air emissions were not quantified.

Because solvent extractables analysis is a cheap, relative easy measurement, the possibility of using solvent extractables removal as a surrogate parameter to predict removal of polycyclic aromatic hydrocarbons was considered. If correlation between SE removal and PAH removal were high, a single measurement could replace 16 individual measurements for estimating PAH removal. Using the calculation protocol in 6.2.2.2, the correlation coefficient relating SE removal and total PAH removal was 0.77. This value suggests that use of SE removal as a surrogate parameter for PAH removal may be possible. Since 80 percent of solvent extractables are unaccounted for in the material balance, however, this concept requires further investigation.

### 3.1.6 Total Organic Carbon (TOC)

Total organic carbon is a surrogate parameter that includes all organic carbon oxidizable by persulfate in the presence of UV light. The oxidized carbon is thus quantified by measuring the evolved carbon dioxide. Given this definition, it is clear that the total organic carbon (TOC) measured in the sample represents the carbon from: SE, PAHs, PCBs, pesticides, other (unidentified) semivolatiles, and volatiles (in untreated material). In addition, some portion of the naturally occurring humic and fulvic acids will oxidize during the time of the analytical test run, and this portion of the organic acids will contribute to total measured carbon. (While humic and fulvic acids are theoretically all oxidizable, the typical Dohrmann instrument will "time out" before the oxidation is complete). Effect of thermal desorption on TOC is given in Tables 30A and 30B. A graph of TOC concentration versus final solids temperature is given in Figure 16. (Error bars shown are for standard error of the mean.)

Removals of TOC ranged from 5 percent to 35 percent, indicating that most of the compounds contributing to TOC (Table 31) were not removed at the process temperatures employed. TOC was examined as a potentially predictive surrogate parameter for several classes of organic compounds. A reasonably large correlation coefficient relates removal of HMW PAHs and TOC removal, as evidenced by the following correlation coefficients ( $r$ ). Caution must be exercised in using TOC removal to predict HMW PAH removal, however, because 85 percent of the PAHs were removed with only 25 percent of the TOC.

TABLE 29  
SUMMARY OF CALCULATIONS  
SOLVENT EXTRACTABLES

BIN	GRAMS IN	<--- GRAMS OUT --->		PERCENT REMOVED
		RESIDUE	CONDENSATE	
A2	1152	163	113	85.9
A3	1208	349	NO DATA	71.2
B1	1184	229	8	80.7
B2	1137	218	12.5	80.8
C1	818	224	0	72.6
C2	883	254	0	71.2
C3	1808	1500	0	17.1
D1	734	253	0	<u>65.5</u>
			AVG	<u>68.1</u>

TABLE 30A  
TOTAL ORGANIC CARBON IN SEDIMENT  
(mg/g Dry Weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	18.6	18.6	18.4	18.4	17.7	17.7	17.7	17.6
After Screening (2s)	18.4	19.0	18.2	18.7	18.0	17.7	18.4	18.2
Before Thermal Proc. (3s)	18.6	19.0	18.9	19.1	18.3	18.0	17.5	18.5
After Thermal Proc. (4s)	12.1	14.7	14.2	10.3	15.2	15.4	16.6	13.2
Solids from 1st Cyclone (7s)	14.8	17.2	17.1	15.9	20.6	16.6	20.5	18.4
Solids from 2nd Cyclone (8s)	10.4	14.7	16.9	15.3	14.6	N/A	15.9	15.3

TABLE 30B  
TOTAL ORGANIC CARBON IN CONDENSATE  
(mg/g liquid)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
After 1st Condenser (9L)	1.58	1.58	.85	.90	.83	.72	.43	.70
After 2nd Condenser (10L)	.85	.51	.53	.75	.28	.25	.17	.37

\* Process conditions same as for sediment

N/A Not Analyzed

Highlighted values are averages of multiple measurements. Averages were calculated using 3-5 individual measurements.

DATA SOURCE: Referenced Battelle Report

FIGURE 16  
TOC (mg/g SOLIDS) VERSUS EXIT TEMPERATURE OF SOLIDS  
(NUMBERS IN PARENTHESES ARE NUMBERS OF DATA POINTS  
FROM WHICH THE STANDARD ERRORS WERE CALCULATED)

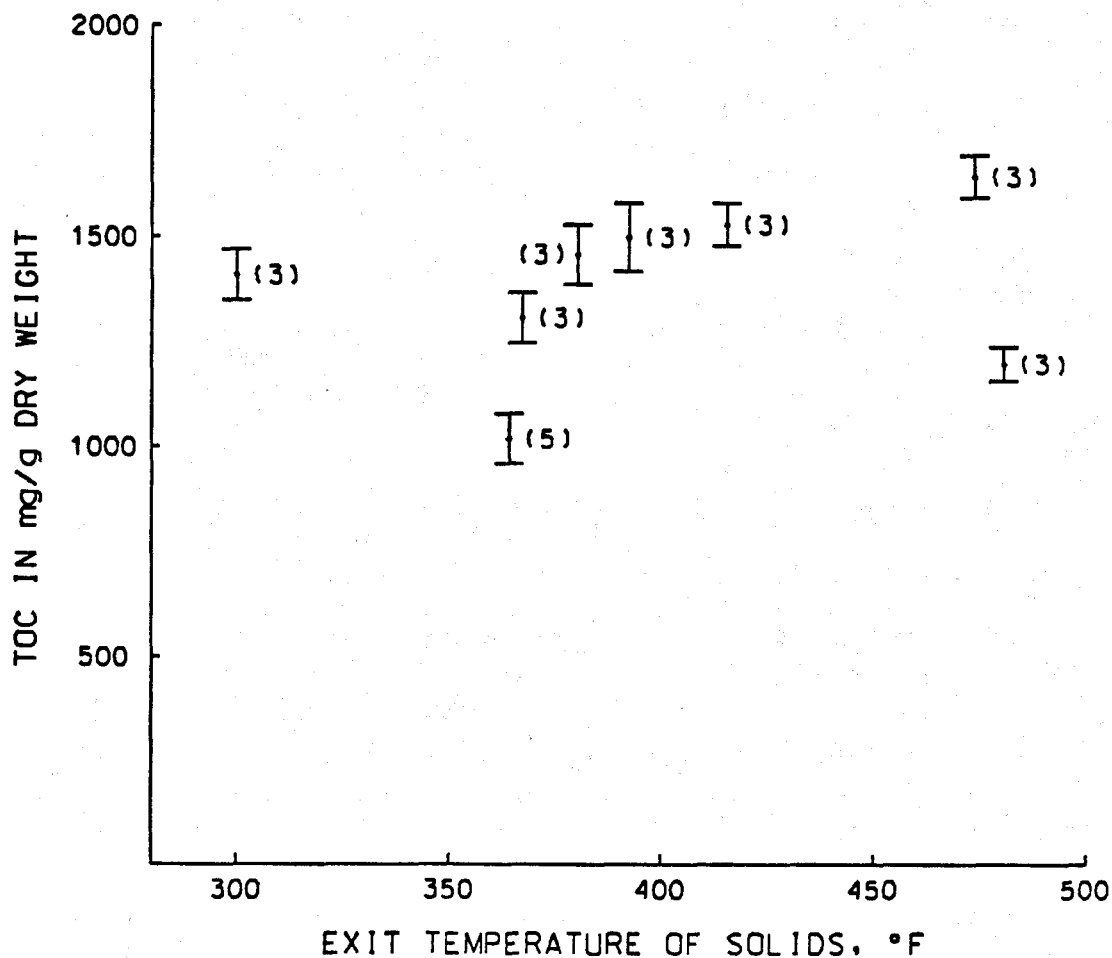


TABLE 31  
SUMMARY OF CALCULATIONS  
TOC

<----- LBS OUT ----->				
<u>BIN</u>	<u>LBS IN</u>	<u>RESIDUE</u>	<u>CONDENSATE</u>	<u>PERCENT REMOVED</u>
A2	18.7	12.1	1.4	35.3
A3	23.3	12.5	0.4	33.5
B1	18.9	14.2	NO DATA	24.9
B2	19.1	10.2	0.3	46.6
C1	18.3	15.2	0.5	16.9
C2	18.0	15.4	0.4	14.4
C3	17.5	16.6	0.3	5.1
D1	18.5	13.2	0.5	<u>28.7</u>
<u>AVG</u>				<u>25.7</u>

<u>Organic Material</u>	<u>r</u>
LMW PAHs	0.58
HMW PAHs	0.81
Total PAHs	0.77
Solvent Extractables	0.71

Fate of TOC is not discussed here, as the "fate" of a surrogate parameter is not a meaningful concept. What should be emphasized is that it was possible to account for an average of 77 percent of the initial TOC and that most of it remained with the sediment.

### 3.1.7 Polychlorinated Biphenyls (PCB)

Analyses were performed before and after processing for four commercial mixtures of PCB (Aroclors): 1242, 1248, 1254, 1260. These are the Aroclors that could be present in the Buffalo River based on site history, thus a total of the individual concentrations of these Aroclors is considered to be total PCB. Total PCB data are presented in Tables 32A and 32B. Data in Tables 32A and 32B are limited primarily to information associated with performance of the thermal processor itself; data associated with more remote parts of the process were not analyzed (N/A) due to cost constraints.

Percent PCB removals were calculated using data in Table 32, and these results are presented in Table 33. Removals ranged from 0 to 100 percent. No correlation was observed between maximum sediment temperature and PCB removal ( $r = -0.05$ ).

TABLE 32A  
TOTAL PCB IN SEDIMENT  
(ng/g dry weight)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
PROCESS CONDITIONS								
Feed Rate lbs/hr	346	716	860	644	484	635	862	388
Residence Time min.	60	30	45	60	90	60	45	90
Max. Sediment Temp. °F	480	380	300	364	392	415	474	367
SAMPLE POINTS								
After Dredging (1s)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
After Screening (2s)	N/A	N/A	ND	N/A	113	N/A	N/A	N/A
Before Thermal Proc. (3s)	ND	ND	184	351	232	209	163	286
After Thermal Proc. (4s)	ND	ND	94	319	17	122	109	ND
Solids from 1st Cyclone (7s)	ND	402	589	158	N/A	284	738	674
Solids from 2nd Cyclone (8s)	ND	67	156	ND	N/A	N/A	ND	N/A

TABLE 32B  
TOTAL PCB IN CONDENSATE  
(ng/L)

Run Number	A2	A3	B1	B2	C1	C2	C3	D1
SAMPLE POINTS *								
Liq. from 1st Condenser (9L)	24698	27343	N/A	ND	25956	39269	5814	ND
Liq. from 2nd Condenser (10L)	66186	69074	27605	ND	12692	17890	19471	ND

N/A - Not Analyzed

ND - None Detected

\* Process conditions same as for sediment

Highlighted values are averages of multiple (3-5) measurements.

DATA SOURCE: Referenced Battelle Report



TABLE 33  
SUMMARY OF CALCULATIONS  
PCB IN SOLIDS

BIN	MG IN	<----- MG OUT ----->		PERCENT REMOVED
		RESIDUE	CONDENSATE	
A2	ND	ND	ND	--
A3	ND	ND	ND	--
B1	63.5	35.3	1.4	44.4
B2	189.2	267.3	0.2	0.0
C1	90.0	6.7	N/A	92.6
C2	91.8	52.8	0.6	42.5
C3	69.3	44.8	2.6	35.4
D1	87.8	0	1.1	100.0

### 3.1.8 Solidification/Stabilization of Treated Residue

Following application of the RETEC thermal desorption technology, the U.S. Army Corps of Engineers solidified selected batches of treated sediment residue with cement. The intent of this action was to curtail mobility of the pollutants remaining in the residue. Four distinct ratios of cement-to-residue ratios were employed: 0.1, 0.2, 0.4, and 0.6. Solidified residues were cured for approximately one month and then tested to determine unconfined compressive strength and degree of pollutant attenuation for Cr, Cu, Hg, Pb, and TOC as measured by either the toxicity characteristic leach procedure (TCLP) or the sequential batch leach test (SBLT). (TCLP is performed in a single leach step while SBLT is performed using 4 sequential extractions.) Results of this work appear in Tables 34 through 36.

From these tests it is concluded that unconfined compressive strength varied directly as cement: residue ratio, indeed the correlation coefficient relating the two variables was 0.994. Additionally, assessments of results for TCLP and SBLT follow:

#### TCLP:

- . Because treated, unsolidified sediment contained no mercury, it was not possible to determine whether solidification could attenuate the mobility of mercury.

- . Solidification of the treated residue resulted in an 89 percent reduction in extract Pb concentration. Reduction was not correlated with cement: residue ratio.

TABLE 34  
SOLIDIFICATION/STABILIZATION UNCONFINED  
COMPRESSIVE STRENGTH (UCS) RESULTS

Run No.	Cement to Residue Ratio	USC (lbs/sq in.)	Average (lbs/sq in.)	Std. Dev. (lbs/sq in.)	Coefficient of Variation (Percent)
B1-B3	0.1	208	250	77	30.7
B1-B3	0.1	203			
B1-B3	0.1	338			
A2	0.2	433	388	48	12.3
A2	0.2	410			
A2	0.2	323			
A2	0.2	384			
A3	0.4	925	898	59	6.6
A3	0.4	836			
A3	0.4	968			
A3	0.4	865			
B2	0.6	1277	1212	86	7.1
B2	0.6	1115			
B2	0.6	1245			

DATA SOURCE: Corps of Engineers Waterways Experiment Station

TABLE 35  
RESULTS OF RESIDUE STABILIZATION

CONCENTRATIONS OF EXTRACTS FROM  
TOXICITY CHARACTERISTIC LEACH PROCEDURE (TCLP)

	BIN	CEMENT TO RESIDUE RATIO	CONCENTRATION IN ug/L				1b/IN <sup>2</sup> UCS	pH
			Cr	Cu	Hg	Pb		
BEFORE STABILIZATION	A2		0.27	4.2	ND	2.57		6.21
	A3		0.71	15.1	ND	6.55		6.10
	B2		0.13	2.4	ND	1.93		6.23
	B1/B3		0.69	13.6	ND	6.16		6.10
AFTER STABILIZATION	A2	0.2	36.5	42.3	ND	ND	388	10.94
	A3	0.4	44.4	12.0	ND	ND	898	11.67
	B2	0.6	17.9	13.1	ND	.72	1212	12.06
	B1/B3	0.1	2.0	30.8	.008	1.07	250	6.61

DATA SOURCE: Referenced Battelle Report

TABLE 36  
SEQUENTIAL BATCH LEACH TEST (SBLT) FOR METALS

CEMENT TO RESIDUE		CR (ug/L)			CU (ug/L)			Hg (ug/L)			Pb (ug/L)		
		Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value	Avg. Value
BIN	RATIO	1st FR.	Σ FR. 2-4	1st FR.	Σ FR. 2-4	1st FR.	Σ FR. 2-4	1st FR.	Σ FR. 2-4	1st FR.	Σ FR. 2-4	1st FR.	Σ FR. 2-4
BEFORE STABILIZATION	A2	0.4	0.8	1.1	5.2	ND	0.00101	ND	0.00101	ND	0.00101	ND	3.4
	A3	1.0	0.7	20.1	8.7	0.00149	0.00356	0.00149	0.00356	ND	0.00356	ND	9.3
	B2	ND	ND	2.4	ND	0.0047	ND	0.0047	ND	ND	ND	ND	ND
	B1/B3	0.6	1.9	4.7	10.4	0.00054	0.00320	0.00054	0.00320	ND	0.00320	ND	7.2
AFTER STABILIZATION	A2	0.2	22.4	60.2	83.7	0.00101	0.00204	0.00101	0.00204	8.1	0.00204	8.1	11.0
	A3	0.4	12.2	34.9	15.1	0.00030	0.00126	0.00030	0.00126	7.1	0.00126	7.1	15.1
	B2	0.6	11.8	44.7	16.9	0.00026	0.00094	0.00026	0.00094	6.8	0.00094	6.8	16.7
	B1/B3	0.1	6.1	12.1	234.2	0.00104	ND	0.00104	ND	13.4	ND	13.4	8.2
CHANGE	A2	0.2	+8.0	+21.6	+78.5	+0.00101	+0.00103	+0.00101	+0.00103	8.1	+0.00103	8.1	+7.6
	A3	0.4	+11.2	+34.2	+8.6	-0.00419	-0.00230	-0.00419	-0.00230	7.1	-0.00230	7.1	+5.8
	B2	0.6	+11.8	+44.7	+24.5	-0.00021	+0.00094	-0.00021	+0.00094	6.8	+0.00094	6.8	+16.7
	B1/B3	0.1	+5.5	+10.2	+223.8	+0.00050	-0.00320	+0.00050	-0.00320	13.4	-0.00320	13.4	+1.0

DATA SOURCE: Referenced Battelle Report

. Leachability of Cu and Cr were increased by solidification. Leachability is not correlated with cement: residue ratio, thus the cause of increased Cu and Cr leaching in solidified material is unknown.

#### SBLT:

. Attenuation of TOC mobility in solidified residue is variable, as measured by Steps 1-4 of the SBLT leach test. These results are shown graphically in Figure 17. In two cases mobility was attenuated and in two cases it was enhanced. These results do not correlate with cement: residue ratio, thus the reason for this pattern is unknown.

. Mobility of all metals but mercury was generally enhanced by the stabilization procedure. Mercury results were variable.

### 3.2 FULL SCALE IMPLEMENTATION

The following discussion provides a description and cost estimate for a full scale remediation of contaminated sediments using the thermal desorption process. Two separate cost estimates were made for sediment remediation involving 10,000 and 100,000 cubic yards of material. These quantities were believed to represent feasible cleanup scenarios for areas with heavily polluted sediments. Due to the anticipated high cost per cubic yard of such a cleanup it did not seem reasonable that millions of cubic yards or several 100's of thousands of cubic yards would be remediated using the thermal desorption process.

#### 3.2.1 Thermal Desorption Remediation

The following discussion provides a description of the thermal desorption system and a cost estimate for the remediation of 10,000 and 100,000 cubic yards of contaminated sediments. In preparing the estimates for sediment treatment, it was assumed that all processing would be conducted at a centralized facility on-site and that the moisture content of the sediments would be adjusted to approximately 60 percent by weight to facilitate handling and treatment operations.

The application of the thermal desorption technology provides for the volatilized components to be condensed and separated into concentrated streams. The aqueous condensate would be treated on-site and combined with the treated solids for dust control, while the concentrated organic condensate would be transported off-site for disposal in a permitted facility.

##### 3.2.1.1 Full-Scale Treatment System--

A nominal processing rate of approximately 41 tons per day (34 cubic yards at 1.2 tons per yard) was assumed. The processing system would have a rated capacity of 2.0 tons per hour for a three shift-per-day operation. A system utilization rate of 85 percent was assumed to provide time for routine maintenance and repair. The proposed throughput assumes a solids residence time of 120 minutes for the full-scale remediation based on the results of the pilot scale demonstration which

FIGURE 17  
RESULTS OF RESIDUE STABILIZATION: TOC ANALYSIS  
OF EXTRACTS FROM SEQUENTIAL BATCH LEACH TEST (SBLT)

FIGURE 17A: BIN A2 CEMENT: RESIDUE RATIO = 0.2

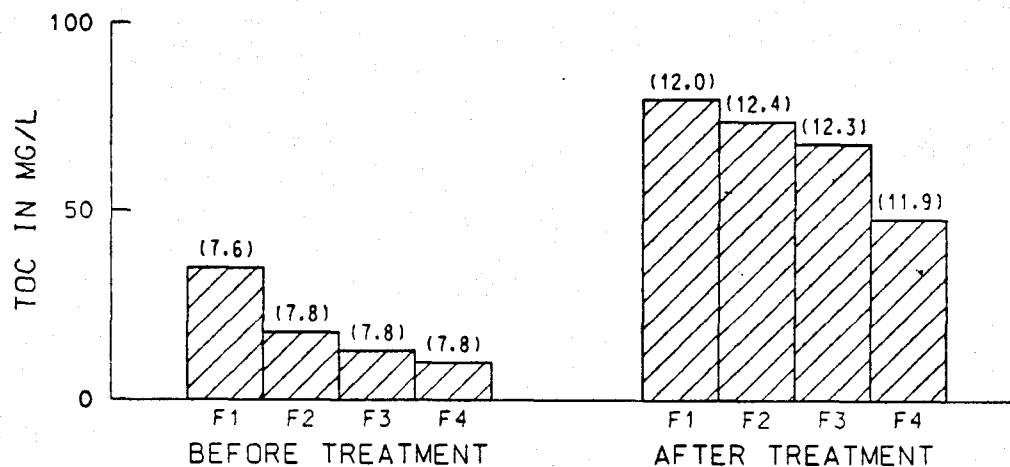
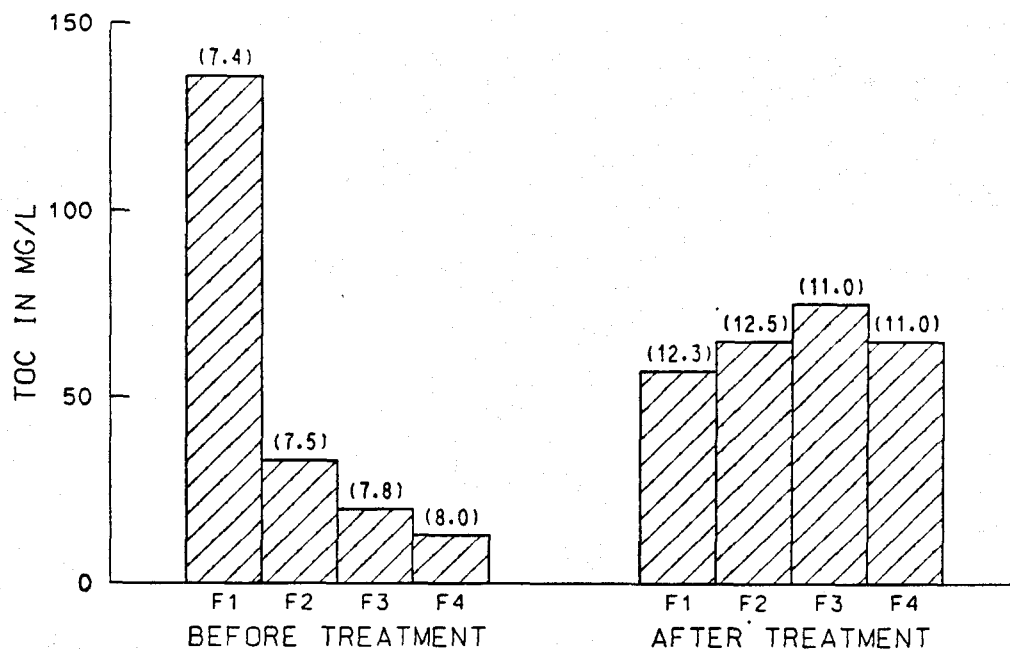


FIGURE 17B: BIN A3 CEMENT: RESIDUE RATIO = 0.4



F# = FRACTION NUMBER  
NUMBER IN PARENTHESIS IS PH OF ASSOCIATED LEACHATE

FIGURE 17C: BIN B2 CEMENT: RESIDUE RATIO = 0.6

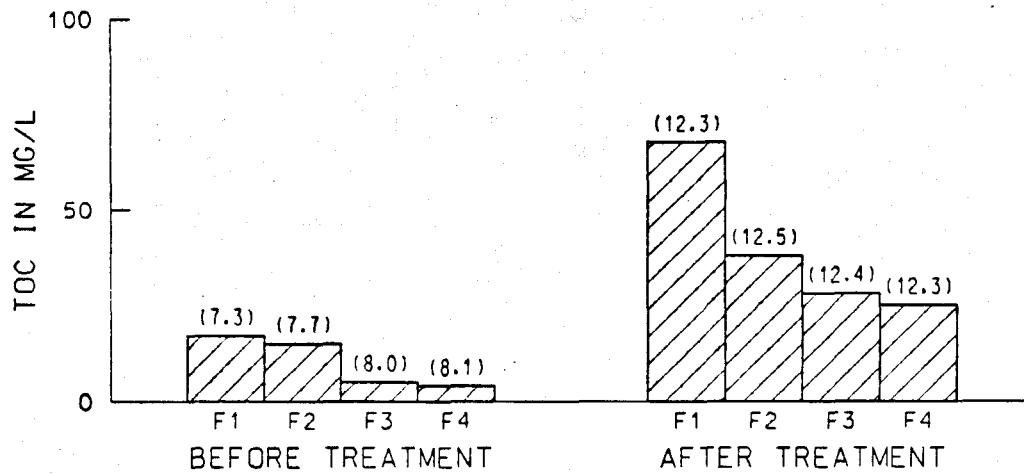
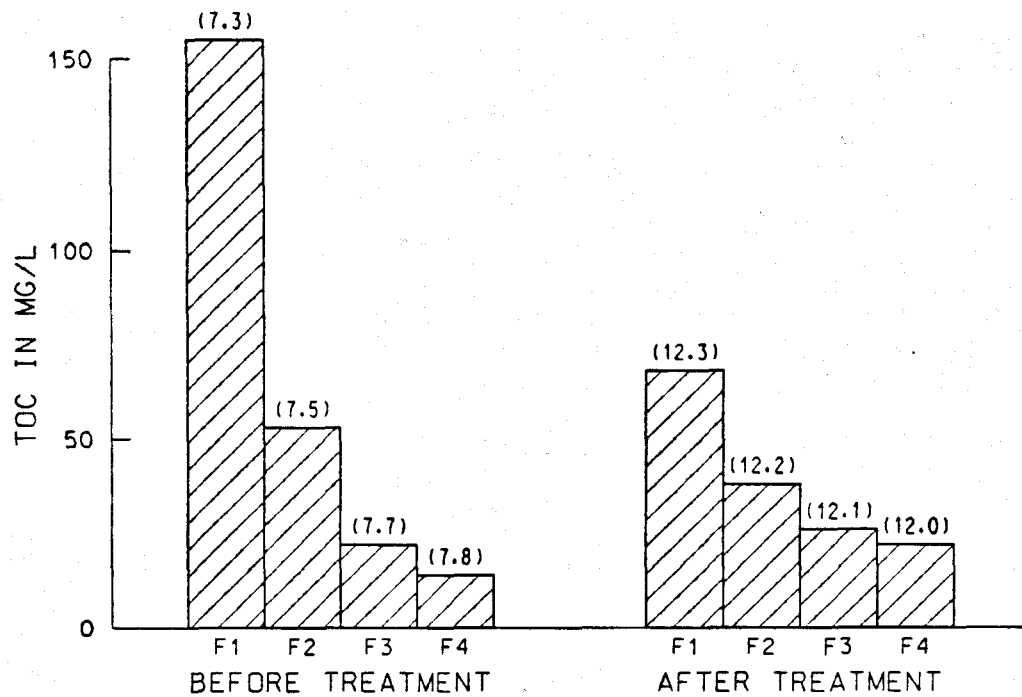


FIGURE 17D: BIN B1/B3 CEMENT: RESIDUE RATIO = 0.1



F# = FRACTION NUMBER  
 NUMBER IN PARENTHESIS IS PH OF ASSOCIATED LEACHATE

indicated that the extended residence would be required to ensure the removal of organic contaminants.

. **Material Feed** - The moisture content of the dredged material would be adjusted to approximately 60 percent prior to screening and treatment. The addition of water and associated mixing would be conducted on a semi-continuous basis. Agitation of the material would be conducted using propeller agitator mixers. Aqueous condensate from the process would be used for make-up water. The sediments would then be pumped from the tanks to the processor at a flow consistent with the proposed processing rate, approximately 6 gallons per minute. The inlet pump line and the pump effluent would be screened to exclude over-sized material or debris. Accumulated oversized material would be removed from the bottom of the tanks during routine maintenance. A positive displacement-style pump would be utilized in transferring the sediments from the tank to the treatment unit.

. **Thermal Processor** - The initial processor in the system is designed to remove moisture and organics from the feed material and reduce the volume of the material for subsequent disposal. A model D-24-24 screw dryer manufactured by the Denver Equipment Company, with a capacity of 2.0 tons per hour, would be used in the processor. The Denver processor uses a contained, non-contact circulating heat transfer fluid to raise the temperature of the solids being treated. Based on results of the pilot scale demonstration, it was assumed that final solids temperatures of 600 to 700° F would be required to ensure the complete removal of organics. The heat transfer fluid for the full scale remediation would be COASTAL HI-TEC salt, which has a maximum operating temperature of 1,100° F. The heat transfer media would be heated with an oil fired system having a capacity to circulate fluid at a rate of 200 gallons per minute (gpm) and provide approximately 3.0 million BTU/per hour to the processor (Remediation Technologies, Inc., 1992).

The final component of the system would be designed to cool the solids to a temperature of 140° F for subsequent handling. The cooling screw would require approximately 20 gpm of cooling water.

It is believed that the build up of feed material around the thermal processor augers could be prevented during a full scale operation by maintaining a feed moisture content of approximately 60 percent. If it were found that the caking of material persisted, the Holo-Flite Screw Processor System could be replaced with a system that continuously self cleans the internal surfaces of the processor.

The off-gas handling system would consist of a cyclone, quench chamber, indirectly-cooled condenser and activated carbon beds. The off gas control system would be designed to accommodate an off-gas flow rate of approximately 2500 cubic feet per minute at 600° F, and "worst case" moisture and organic loading of 2400 pounds per hour and 4 pounds per hour respectively. A cyclone particulate removal system would remove any fine solid particles (>10 um) which may be entrained with the off-gases.

These solids would be removed and combined with the feed material for reprocessing. The volatilized organics and moisture would be condensed into a combined liquid stream. The condenser would be designed to achieve 90 percent removal of organics and moisture in the gas. Separation of the organics and aqueous condensate streams would be enhanced by the use of a coalescing plate separator. The condensed organics would be disposed of off site.

Approximately 5 gpm of aqueous condensate would be generated as a result of treatment operations. One half of the condensate would be used to dilute the dredged sediments in the mixing tanks, while the remaining volume of condensed water would be treated on site using biological treatment or activated carbon. After the liquid and particulate are removed from the purge gas stream the gas would be treated to remove residual volatile organics. Treatment would consist of filtration through granulated activated carbon beds.

### 3.2.1.2 Cost Estimate for Sediment Remediation--

Cost estimates for the remediation of 10,000 and 100,000 cubic yards of contaminated sediment using the thermal desorption technology were prepared. The estimated operating costs (+/- 20 percent) for treating the contaminated sediments on site are shown in Tables 36 and 37. Unit prices were determined using a processing rate of 890 tons (612 cubic yards of "as-dredged" material (40 percent moisture) per month, assuming a 24 hour per day operation with an 85 percent system utilization rate. The estimate includes costs for all mobilization/demobilization, thermal treatment, associated analytical activities, on-site treatment of the aqueous condensate, and the off-site disposal of the organic condensate stream. Costs associated with sediment excavation and transport to the treatment site were not included. These costs can be expected to be less than 10 percent of the associated remediation cost discussed below. Engineering and design work would add approximately 10 percent to the project cost, as would construction management. Therefore, total project cost would be roughly 1-1/3 times the remediation cost shown below.

. Remediation of 10,000 Cubic Yards - Treatment costs for processing this volume of waste were estimated at \$535 per cubic yard, with the work being completed in approximately 16 months. Mobilization and demobilization costs were estimated to be \$500,000 while the monthly equipment charge was estimated at \$132,000. The equipment charge was calculated to provide a 25 percent return on capital investment over the duration of the contract.

Field labor and utilities were estimated to make up approximately 30 percent of the total treatment cost. The labor costs includes 4 workers per shift and three shifts per day at an average hourly rate of \$35, including overhead. Utility costs are based upon the fuel requirements to maintain the temperature of the heat transfer media during treatment, and the electrical requirements of the system (200 kw, 480 v, 225 amp).

Maintenance and activated carbon disposal comprise less than 5 percent of the cost. Activated carbon disposal/replacement was estimated at \$1000 per month. Carbon would be recharged and



reused until its useful life was exhausted. Spent carbon would be disposed of by landfilling or incineration if required. The off-site disposal of the condensed liquid organics was conservatively estimated at \$1000 per ton while the on-site treatment of the aqueous condensate was estimated to cost \$0.02 per gallon. This cost equates to approximately \$8 per ton of

dredged sediment. The total cost for processing 10,000 cubic yards of sediments was estimated at \$5,350,000 and is shown in Table 37.

. Remediation of 100,000 Cubic Yards - The cost estimate for treating 100,000 yards of contaminated sediments includes the use of four parallel treatment systems to allow the completion of the remediation in a reasonable time period. As discussed previously, each system would process 612 cubic yards of "as-dredged" sediments per month, assuming a 24 hour per day operation with an 85 percent system utilization rate. With this treatment train, the sediment remediation work could be completed within 41 months at an estimated unit cost of \$352 per cubic yard. Mobilization and demobilization costs were estimated to be \$1,200,000, or \$12 per cubic yard of sediment. Other operating expenses such as fuel, maintenance and waste disposal will have unit costs similar to those discussed under the smaller treatment scenario.

Monthly equipment charges for the four treatment units were estimated to be \$363,000, resulting in a monthly charge per individual unit that is significantly less than the monthly charge for the single unit anticipated for the remediation of 10,000 cubic yards of sediment. This is due to the increased period of time, 41 verses 16 months, available to recover the capital investment. The total cost for processing 100,000 cubic yards of contaminated sediments was estimated to be \$35,200,000 and is shown in Table 38.

Variations in the moisture content can impact the thermal requirements, while variations in the organic content of the feed sediment would primarily affect the pricing of services by changing the rate of activated carbon usage. RETEC does not believe that any of these factors or material processing requirements would significantly affect the throughput of the system or the estimated cost of treatment (Remediation Technologies, Inc., 1992).

### 3.3 CONCLUSIONS AND RECOMMENDATIONS

#### 3.3.1 Conclusions

A review of the results from the program provides the following conclusions related to material composition, material handling, effectiveness of treatment, and process operation.

1. Material Handling - The dredged materials were relatively free of debris or over-sized material which would adversely affect the processing of the sediments during full-scale implementation. Some problems were caused by the physical

TABLE 37

## COST ESTIMATE FOR REMEDIATING 10,000 CUBIC YARDS OF SEDIMENT

<u>DESCRIPTION</u>	<u>ESTIMATED COST</u>
Mobilization and Demobilization (Treatment)	\$ 500,000
Equipment Rental	2,150,000
Utilities: Fuel	250,000
Electricity: Cooling	320,000
Misc.	160,000
Operating Field Labor	1,630,000
Maintenance	80,000
Activated Carbon	20,000
Other Operating Costs	160,000
Disposal of Organic Condensate and Treatment of Aqueous Condensate	80,000
TOTAL	\$5,350,000
TOTAL COST PER CUBIC YARD	\$ 535

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DATA SOURCE: Referenced RETEC, Inc. Report

TABLE 38

## COST ESTIMATE FOR REMEDIATING 100,000 CUBIC YARDS OF SEDIMENT

<u>DESCRIPTION</u>	<u>ESTIMATED COST</u>
Mobilization and Demobilization	\$ 1,200,000
Equipment Rental	14,900,000
Utilities: Fuel	2,500,000
Electricity: Cooling	3,200,000
Misc.	1,000,000
Operating Field Labor	9,600,000
Maintenance	800,000
Activated Carbon	200,000
Other Operating Costs	1,000,000
Disposal of Organic Condensate and Treatment of Aqueous Condensate	800,000
TOTAL	\$35,200,000
TOTAL COST PER CUBIC YARD	\$352

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DATA SOURCE: Referenced RETEC, Inc. Report and Corps of  
Engineers, Buffalo District

characteristics of the material: (1) Sediments can cause conveyance problems for solids handling systems such as conveyors, bucket elevators, and screw conveyors due to their cohesive properties. However, the feed material was determined to be readily pumpable at moisture contents above 45 percent by weight. (2) Dried sediments can collect in the processor, eventually preventing the rotation of the screw auger. Results from the pilot scale demonstration indicate that this problem was more pronounced when feed material with lower moisture contents were being processed. Minimal problems were encountered when processing material with a moisture content in excess of 50 percent by weight. From the limited data available it was not clear if this was due to a physical change in the properties of the solids or is the result of a lower mass feed rate of solids due to dilution with water.

2. Process Operations - After initial optimization of the material handling system, the technology operated without significant problems and provided the following information. In general, the heat transfer characteristics for the sediment were low, resulting in exit solids temperatures that were significantly lower than anticipated, 300 to 535°F versus 700°F. Residence times of 60 minutes were appropriate to achieve moisture removal/mass reduction, while consistent organic removal required treatment for periods in excess of 90 minutes. Particulate buildup in the off-gas control system was not a significant problem due to the high moisture content of the feed material. One of the operating results was the poor separation of the organic and aqueous condensates due to the dilute nature of the oil stream. RETEC attempted to separate the streams by "controlled condensation," separation on the basis of condensation temperature. This proved to be largely unsuccessful, probably due to the low concentration of organics versus moisture in the waste. Results from several test runs indicated that the aqueous and organic streams generally contained low concentrations of volatile organics which were concentrated to some degree in the organic stream. Effective separation of such a dilute stream would require the use of a physical separator.

3. Feed material containing 44-56 percent solids was successfully pumped and dried to a solids content  $\geq$  95 percent.

4. Removal levels for constituents of concern were contaminant specific. Ranges of removal for each constituent of concern were as follows:

- a. Mercury: 9-100%
- b. Low molecular weight polycyclic aromatic hydrocarbons: 45-90%
- c. High molecular weight polycyclic aromatic hydrocarbons: 42-96%
- d. Oil and grease: 17-86%
- e. Total organic carbon (TOC): 5-35%

- f. Polychlorinated biphenyls (PCBs): 0-100%
- g. Chromium: 0-9%
- h. Copper: 0-11%
- i. Lead: 0-13%

5. Removal of organic materials was not strongly correlated with measured process conditions such as maximum sediment temperature, residence time, and percent moisture in the feed. Some other parameter is controlling removal.

6. Since copper, chromium, and lead remain with the treated sediment, the dried sediment is a potential candidate for remediation by a stabilization/solidification technology. While stabilization/solidification with a cementitious process was not successful for chromium and copper when the material was ground up, it is possible that chromium and copper leachability would be negligible if the unground material was tested by exposure to normal weathering processes.

7. Solvent extractables and "TOC" are both potentially usable as surrogate parameters for predicting removal of high molecular weight PAHs.

8. Air emissions were measured for PAHs, dioxins, furans, and PCBs, and were extremely low:

Low molecular weight PAHs: 1.6 - 18.0 mg/hour  
High molecular weight PAHs: 0.4 - 8.6 mg/hour  
Dioxins: 0.005 - 0.017 mg/hour  
Furans: 0.000 - 0.032 mg/hour  
PCBs: 1.3 - 2.4 mg/hour

9. It is of interest to determine the percent of each hazardous material entering the processor that was released to the atmosphere via air emissions. This value was quantifiable for PAHs only;  $\leq 0.004$  percent of the PAHs in the feed material were discharged via air emissions.

10. Stabilization/solidification of the treated residue resulted in an 89 percent attenuation of the mobility of lead. Leachability of copper and chromium were increased.

11. Fate of the removed materials was not determined in many cases. Factors contributing to this problem were (1) the combining of stream rates rather than taking individual weights, (2) slight particulate losses at the processor seals, (3) processing of relatively clean sediment, (4) exclusion of metals analysis in the air sampling, and (5) failure to quantify materials captured by carbon.

### 3.3.2 Recommendations/Lessons Learned

The following recommendations are made for performing future pilot studies in thermal desorption.

1. Operation at higher temperatures should be considered to

assess whether higher removal levels can be achieved for semi-volatiles and other organic materials.

2. Individual weights of each stream should be obtained rather than combined weights (e.g., Solids from each cyclone should be weighed and recorded, as should the treated residue. The solids should not all be combined and then reported as a single weight.). The taking of individual weights will make it possible to determine the fate of each contaminant.

3. Analysis of air emissions should include analysis for metals.

4. Sediments with greater contaminant levels should be selected for treatment. A large percent error is inherent in measurements and mass balances for low concentrations of contaminants.

5. Use of solvent extractables and/or "Total Organic Carbon" as surrogate parameters for estimating removal of high molecular weight PAHs should be further investigated.

6. Analysis of activated carbon for organics and metals should be considered and carried out. PAHs and PCBs are likely partitioning to the carbon in the emissions system, because of the fundamental nature of activated carbon to adsorb organics.

3.3.3 The following recommendations concern the use of the thermal desorption and solidification for sediment remediation:

1. Thermal desorption should be tried again at higher temperatures/longer residence times to achieve a more complete assessment of organic contaminant removal ability is made.

2. Thermal desorption should be applied to a more contaminated feed material than the Buffalo River sediments before an assessment of organic contaminant removal ability is made.

3. Solidification/stabilization is not practical for Buffalo River residues, as two of the three metal contaminants (Cu, Cr) leach more quickly when solidified than when the material has not been treated in this way.

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**APPENDIX A**  
**SAMPLE CALCULATIONS**





SAMPLE MASS BALANCE CALCULATION  
MASS BALANCE FOR CHROMIUM

PART A: ADJUSTMENT OF STREAM WEIGHTS TO DRY WEIGHT BASIS:

A1: NO DATA

A2:

INPUT

$$(2010 \text{ lbs}) \times \left( \frac{.554 \text{ lbs dry solids}}{1 \text{ b}} \right) \times \left( \frac{.454 \text{ kg}}{1 \text{ b}} \right) = 505 \text{ kg dry solids}$$

OUTPUT SOLIDS

$$\begin{aligned} \text{Average \% solids in treated solids + cyclone residue} = \\ (1055 \text{ lb}) \times (.998) \times (0.454) = 478 \text{ kg} \end{aligned}$$

OUTPUT LIQUIDS

$$(890 \text{ lb}) \times (.011 \text{ lb solids/lb liquid}) \times (0.454) = 4.4 \text{ kg}$$

A3:

$$\text{INPUT } (2150)(.550)(.454) = 537 \text{ kg}$$

$$\text{OUTPUT SOLIDS } (1235)(.995)(.454) = 557 \text{ kg}$$

$$\text{OUTPUT LIQUIDS } (688)(.008)(.454) = 2.5 \text{ kg}$$

B1

$$\text{INPUT } (1355)(.561)(.454) = 345$$

$$\text{OUTPUT SOLIDS } (845)(.986)(.454) = 378$$

$$\text{OUTPUT LIQUIDS } (620)(.008)(.454) = 2.3$$

B2

$$\text{INPUT } (2175)(.546)(.454) = 539 \text{ kg}$$

$$\text{OUTPUT SOLIDS } (1850)(.998)(.454) = 838 \text{ kg}$$

$$\text{OUTPUT LIQUIDS } (445)(.007)(.454) = 1.4 \text{ kg}$$

C1

INPUT (1640)(.521)(.454) = 388

OUTPUT SOLIDS (880)(.998)(.454) = 398

OUTPUT LIQUIDS (840)(.009)(.454) = 3.4

C2

INPUT (1905)(.508)(.454) = 439

OUTPUT SOLIDS (955)(.999)(.454) = 433

OUTPUT LIQUIDS (880)(.005)(.454) = 2 kg

C3

INPUT (1940)(.483)(.454) = 425

OUTPUT SOLIDS (950)(.952)(.454) = 411

OUTPUT LIQUIDS (850)(.009)(.454) = 3.5

D1

INPUT (1515)(.446)(.454) = 307

OUTPUT SOLIDS (700)(.999)(.454) = 317

OUTPUT LIQUIDS (950)(.004)(.454) = 1.7

#### SOLIDS BALANCE

RUN	SOLIDS INPUT (kg)	SOLIDS OUTPUT (kg)	SOLIDS OUTPUT IN LIQUID (kg)
A2	505	478	4.4
A3	537	(557)	2.5
B1	345	(378)	2.3
B2	539	(838)	1.4
C1	388	(398)	3.4
C2	439	433	2.0
C3	425	411	3.5
D1	307	(317)	1.7

For numbers in parentheses, output exceeds input according to calculations. Possible reasons include material holdup in the equipment and errors inherent in the analytical test methods.

# MASS BALANCES/METALS

Chromium  
(A2)

$$505,000 \text{ g solids } \frac{(47 \times 10^{-6} \text{ g Cr})}{\text{g solids}} = 23.7 \text{ Cr in}$$

Since it is not known what % of total solids came from the cyclones, the following is assumed for these calculations:

cyclone 1: 4%  
cyclone 2: negligible

∴ Overall concentration of processed solids is:

$$.96(70) + .04(137) = 73 \text{ ug/g}$$

IN SOLIDS:

$$(477,000 \text{ g}) \times \frac{(73 \times 10^{-6} \text{ g Cr})}{\text{g}} = 34.8 \text{ g}$$

IN CONDENSATE SOLIDS: Consider this negligible

$$(4,400 \text{ g}) \frac{(137 \times 10^{-6} \text{ g})}{\text{g}} = .60 \text{ g}$$

In Condensate:

$$890 \text{ lbs } \frac{(.454 \text{ kg})}{\text{lb}} = 404 \text{ kg} - 4 \text{ kg solids output from liquid}$$

$$404 \text{ kg } \frac{\text{L}}{\text{kg}} \frac{(13.130 \times 10^{-6} \text{ g Cr})}{\text{L}} = 5.2 \text{ grams}$$

$$\underline{\text{A3: In:}} (537,000)(68)(10^{-6}) = 36.5 \text{ Cr in}$$

$$.96(61) + .04(148) \approx 65 \text{ ug/g}$$

$$\text{Out: } (557,000)(65 \times 10^{-6}) = 36.2 \text{ Cr in treated solids}$$

Out/Liquids: Correction to liquid amount by removing amount solids is negligible and is ignored for this and future calculations.

$$\frac{(688)(454)(14,200 \times 10^{-6})}{(1000)} = 4.4 \text{ g}$$

B1:

$$\text{In: } (345,000)(47)(10^{-6}) = 16.2 \text{ g}$$

$$(0.96)(56) + (0.04)(129) \approx 59 \text{ ug/g}$$

$$\text{Out (solids): } (378,000)(59 \times 10^{-6}) = 22.3 \text{ g}$$

$$\text{Out (liquid): } \frac{(620)(454)(1847 \times 10^{-6})}{(1000)} = 0.5 \text{ g}$$

B2: In:  $(539,000)(58)(10^{-6}) = 31.3 \text{ g}$

$$0.96(44) + 0.04(106) \approx 47 \text{ ug/g}$$

$$\text{Out (solid): } (838,000)(47)(10^{-6}) = 39.4 \text{ g}$$

$$\text{Out (liquid): } \frac{(445)(454)(200)(10^{-6})}{(1,000)} = 0.04 \approx 0 \text{ g}$$

B3: Closure too poor to consider mass balance on Cr.

C1: In:  $(388,000)(73)(10^{-6}) = 28.3 \text{ g Cr in}$

$$0.96(67) + 0.04(136) = 70 \text{ ug/g Cr in treated solids}$$

$$(398,000)(70 \times 10^{-6}) = 27.9 \text{ g}$$

$$\text{Out (liquid): } \frac{(840)(454)(830)(10^{-6})}{1000} = 0.3 \text{ g}$$

C2: In:  $(439,000)(59)(10^{-6}) = 25.9 \text{ Cr in}$

$$.96(58) + .04(129) \approx 61 \text{ ug/g Cr}$$

$$\text{Out: } (433,000)(61)(10^{-6}) = 26.4 \text{ Cr in treated solids}$$

$$\text{Out (liquid): } \frac{(880)(454)(410)(10^{-6})}{1000} = 0.2 \text{ g Cr}$$

C3: In:  $(425,000)(69)(10^{-6}) = 29.3 \text{ Cr in}$

$$0.96(62) + 0.04(140) = 65 \text{ ug/g}$$

Out:  $(411,000)(65)(10^{-6}) = 12.1 \text{ g Cr out with solids}$

Out (Liquid):  $\frac{(454)(850)(1140)(10^{-6})}{(1000)} = 0.4 \text{ g Cr}$

D1: In:  $(307,000)(55)(10^{-6}) = 16.9 \text{ g Cr}$

$$0.96(62) + 0.04(112) = 64 \text{ ug/g}$$

Out:  $(317,000)(64)(10^{-6}) = 20.3 \text{ g Cr in solids}$

Out (liquid):  $\frac{(950)(454)(723)(10^{-6})}{1000} = 0.3$

CORRELATION COEFFICIENT:

$$r = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{[\sum (x_i - \bar{x})^2 \sum (y_i - \bar{y})^2]^{1/2}}$$

WHERE:  $x_i$  = given value of process variable (e.g., temperature)

$y_i$  = given value of measured parameter (e.g., % solids)

$\bar{x}$  = average value of process variable

$\bar{y}$  = average value of measured parameter

# AIR EMISSIONS OF POLYCHLORINATED BIPHENYLS

PCB	Minimum		Maximum	
	LBS/Hour	Grams/Hour	LBS/Hour	Grams/Hour
Monochlorobiphenyl	1.25E-07	5.67E-05	2.23E-07	1.01E-04
Dichlorobiphenyl	1.25E-07	5.67E-05	2.23E-07	1.01E-04
Trichlorobiphenyl	1.25E-07	5.67E-05	2.23E-07	1.01E-04
Tetrachlorobiphenyl	2.49E-07	1.13E-04	4.46E-07	2.02E-04
Pentachlorobiphenyl	2.49E-07	1.13E-04	4.46E-07	2.02E-04
Hexachlorobiphenyl	2.49E-07	1.13E-04	4.46E-07	2.02E-04
Heptachlorobiphenyl	3.82E-07	1.73E-04	6.84E-07	3.11E-04
Octachlorobiphenyl	3.82E-07	1.73E-04	6.84E-07	3.11E-04
Nonachlorobiphenyl	3.82E-07	1.73E-04	6.84E-07	3.11E-04
Decachlorobiphenyl	<u>6.31E-07</u>	<u>2.86E-04</u>	<u>1.13E-06</u>	<u>5.13E-04</u>
TOTAL	2.90E-06	1.32E-03	5.19E-06	2.23E-03

# AIR EMISSIONS OF PARTICULATES

	Minimum		Maximum	
	LBS/Hour	Grams/Hour	LBS/Hour	Grams/Hour
Particulates	0.0019	0.086	0.0026	1.18



# AIR EMISSIONS OF POLYCYCLIC AROMATIC HYDROCARBONS

PAH	Minimum		Maximum	
	LBS/Hour	Grams/Hour	LBS/Hour	Grams/Hour
Naphtahalene	2.35E-06	1.07E-03	2.58E-05	1.17E-02
Acenaphthylene	3.01E-08	1.37E-05	1.26E-06	5.72E-04
Acenaphthene	9.65E-08	4.38E-05	2.49E-06	1.13E-03
Fluorene	1.16E-08	5.27E-06	1.14E-06	5.18E-04
Phenanthrene	9.58E-07	4.35E-05	7.92E-06	3.60E-03
Anthracene	1.04E-07	<u>4.72E-05</u>	9.51E-06	<u>4.32E-03</u>
		1.22E-03		2.18E-02
Fluoranthene	3.89E-07	1.77E-04	2.33E-06	1.06E-03
Pyrene	2.22E-07	1.00E-04	4.56E-06	2.07E-03
Benz(a)anthracene	4.38E-08	1.99E-05	9.16E-06	4.16E-03
Chrysene	5.30E-08	2.40E-05	1.04E-06	4.72E-04
Benzo(bjk)fluoranthrene	9.52E-08	4.32E-05	4.72E-07	2.14E-04
Benzo(a)pyrene	2.63E-08	1.19E-05	5.16E-07	2.34E-04
Indo(1,2,3-cd)pyrene	1.99E-08	9.03E-06	1.28E-07	5.81E-05
Dibenz(a,h)anthracene	1.90E-08	8.63E-06	1.22E-07	5.54E-05
Benzo(g,h,i)perylene	2.70E-08	<u>1.22E-05</u>	5.46E-07	<u>2.48E-04</u>
		405.86E-06		857.15E-05
TOTAL	4.44E-06	2.02E-03	5.86E-05	2.66E-02

## AIR EMISSIONS OF DIOXINS\*

DIOXIN	Minimum		Maximum	
	nanograms/sec	nanograms/hour	nanograms/sec	nanograms/hour
2378-TCDD	7.00E-04	2.52	1.66E-03	5.98
12378-PeCDD	3.46E-04	1.25	2.01E-03	7.24
123478-HxCDD	8.67E-05	0.31	2.68E-04	0.96
123678-HxCDD	5.33E-05	0.19	2.41E-04	0.87
123789-HxCDD	6.67E-05	0.24	2.72E-04	0.98
1234678-HpCDD	1.67E-05	0.06	2.91E-04	1.05
OCDD	1.30E-05	<u>0.04</u>	8.99E-05	<u>0.32</u>
TOTAL		4.61		17.40

\* Emissions reported as 2378-TCDD Toxicity Equivalent (EPA 1989)

# AIR EMISSIONS OF FURANS\*

FURAN	Minimum		Maximum	
	nanograms/sec	nanograms/hour	nanograms/sec	nanograms/hour
2378-TCDF	0.00E+00	0.00	6.08E-05	0.22
12378-PeCDF	0.00E+00	0.00	0.00E+00	0.00
23478-PeCDF	0.00E+00	0.00	5.48E-03	19.73
123478-HxCDF	0.00E+00	0.00	1.30E-03	4.68
123678-HxCDF	1.47E-05	0.05	1.07E-03	3.85
234678-HxCDF	0.00E+00	0.00	7.12E-05	0.26
123789-HxCDF	0.00E+00	0.00	3.38E-04	1.22
1234678-HpCDF	0.00E+00	0.00	3.68E-04	1.32
1234789-HpCDF	0.00E+00	0.00	0.00E+00	0.00
OCDF	0.00E+00	<u>0.00</u>	0.00E+00	<u>0.00</u>
TOTAL		0.05		32.28

\* Emissions reported as 2378-TCDD Toxicity Equivalents (EPA 1989)



**APPENDIX B**

**UNREDUCED ANALYTICAL DATA**



REPORT OF CHEMICAL ANALYSES  
Volume I: Metals and Conventional

PROJECT: Buffalo River Pilot Project

Prepared for:

USEPA Great Lakes National Program Office  
Attn: Dr. Steve Garbaciak  
230 S. Dearborn  
Chicago, IL 60604

## SAMPLE LABEL KEY

Sample label key is explained by using the following examples:

07OCT 12:34 1S A O ← Label  
(1) (2) (3) (4) (5) ← Key

- (1) Date of sample
- (2) Time of sample
- (3) Sampling point number from Figure 10
  - Where S = solid stream
  - L = liquid stream
  - G = gas stream
- (4) Bin (A,B,C, or D: There were 4 bins filled)
- (5) Type of sample, where:
  - O = organic
  - M = metal
  - L = leach test
  - G = grain size
  - S = solids
  - C = Total Organic Carbon (TOC)

When sample has additional number preceding the last letter of the series, i.e.

23OCT 4:28 4S A2 O

The number is a run number, i.e. Run 2 from Bin A

REPORT OF: CHEMICAL ANALYSES

PROJECT: BUFFALO RIVER PILOT PROJECT

DATE:

ISSUED TO: Dr. Steve Garbaciak  
Technical Project Manager  
USEPA Great Lakes National Program Office  
230 S. Dearborn  
Chicago, IL 60604

CF#: 379GLBR

## INTRODUCTION

This report summarizes the results from analyses performed on pilot study samples which were submitted by the U.S. Army Corps of Engineers Great Lakes Division, Buffalo District.

## SAMPLE CUSTODY

Samples were received in good condition from October 7, 1991 through November 25, 1991. Samples were logged in and stored as specified in the narrative accompanying each method description. Samples were analyzed within the holding times specified in the QA plan. Any exceptions are noted in the narrative associated with each analysis.

## GRAIN SIZE

Seventeen samples were analyzed for grain size according to Battelle SOP# MSL-M-37 (a modified version of Plumb; 1981). The samples were separated into four classes: gravel, sand, silt and clay. Those classes (excluding gravel) were further subdivided into 5 sand fractions, 6 silt fractions and 4 clay fractions. Samples were stored at  $4 \pm 2^\circ\text{C}$  prior to and following analysis. Five samples (two in duplicate) were archived and maintained at  $4 \pm 2^\circ\text{C}$  for possible analysis in the future.

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
07OCT1:221SCG	Sediment	Grain Size	379GLBR*25
07OCT12:511SBG	Sediment	Grain Size	379GLBR*31
07OCT12:161SAG	Sediment	Grain Size	379GLBR*32
07OCT1:561SDG	Sediment	Grain Size	379GLBR*33
08OCT5:222SD2G	Sediment	Grain Size	379GLBR*47
09OCT12:002SAG	Sediment	Grain Size	379GLBR*61
09OCT10:502SBG	Sediment	Grain Size	379GLBR*69
09OCT5:082SCG	Sediment	Grain Size	379GLBR*77
23OCT4:304SA2G	Sediment	Grain Size	379GLBR*120
25OCT9:264SA3G	Sediment	Grain Size	379GLBR*156
25OCT2:324SB1G	Sediment	Grain Size	379GLBR*184
31OCT9:274SB2G	Sediment	Grain Size	379GLBR*219
31OCT2:364SB3G	Sediment	Grain Size	379GLBR*241
19NOV8:584SC1G	Sediment	Grain Size	379GLBR*299
19NOV4:464SC2G	Sediment	Grain Size	379GLBR*321
20NOV3:114SC3G	Sediment	Grain Size	379GLBR*346
21NOV5:204SD1G	Sediment	Grain Size	379GLBR*391

## ARCHIVED SAMPLES

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
08OCT5:232SD2G	Sediment	Grain Size	379GLBR*47-T2
08OCT5:242SD2G	Sediment	Grain Size	379GLBR*47-T3
21OCT6:024SA1G	Sediment	Grain Size	379GLBR*94
19NOV4:464SC2G	Sediment	Grain Size	379GLBR*321-T1
19NOV4:464SC2G	Sediment	Grain Size	379GLBR*321-T2
22NOV3:124SD2G	Sediment	Grain Size	379GLBR*420



Some values for % dry weight are greater than 100% due to absorption of moisture during the cooling step of the method for determining % dry weight. If a desiccator had been used, the samples would have exhibited % dry weight values - 99.5%. The analyst did not use a desiccator for the oven-drying step for the determination of grain size. Therefore, when the analyst calculates the data, any effect of moisture absorption is canceled out through the equations resulting in valid data.

## METALS

Forty-six sediment samples and thirty-three water samples were analyzed for metals. The sediment samples were dried (Battelle SOP# MSL-M-3) and an aliquot was analyzed by X-ray fluorescence for Cr, Cu and Pb. A separate dried aliquot was digested with nitric, hydrofluoric and perchloric acids (Battelle SOP# MSL-M-7), then analyzed for Hg by cold vapor atomic absorption (Battelle SOP# MSL-M-31). An aliquot of each water sample was digested with hot nitric acid (Battelle SOP# MSL-M-22) and analyzed by flame atomic absorption and graphite furnace atomic absorption for Cr, Cu and Pb according to Battelle SOP# MSL-M-32 (based on EPA method 200.9). A separate aliquot was digested with a solution of nitric and sulfuric acid, then analyzed for Hg by cold vapor atomic fluorescence following Battelle SOP# MSL-M-11. According to the Buffalo River QA plan we were to analyze the Hg samples following Battelle SOP# MSL-M-27 for Hg in water, however, due to the high content of combustion by-products from organic contaminants it was necessary to treat these samples as sediments. Thirteen sediment samples (3 in quadruplicate) and 12 water samples (2 in quadruplicate, 2 in duplicate) were archived for possible analysis in the future. Sediment samples were stored at  $-70 \pm 10^\circ\text{C}$  prior to drying. Once dried, the samples were stored at room temperature prior to and following analysis. Water samples were acidified at the time of collection and stored at room temperature prior to and following analysis.

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
07OCT1:261SCM	Sediment	Metals	379GLBR*26
07OCT12:191SAM	Sediment	Metals	379GLBR*27
07OCT2:001SDM	Sediment	Metals	379GLBR*28
07OCT12:541SBM	Sediment	Metals	379GLBR*29
08OCT4:572SDM	Sediment	Metals	379GLBR*49
09OCT12:002SAM	Sediment	Metals	379GLBR*60
09OCT10:502SBM	Sediment	Metals	379GLBR*68
09OCT5:052SCM	Sediment	Metals	379GLBR*76
22OCT11:287SA1M	Sediment	Metals	379GLBR*103
22OCT11:358SA1M	Sediment	Metals	379GLBR*106
23OCT1:303SA2M	Sediment	Metals	379GLBR*115
23OCT4:314SA2M	Sediment	Metals	379GLBR*119
24OCT10:457SA2M	Sediment	Metals	379GLBR*135
24OCT10:508SA2M	Sediment	Metals	379GLBR*138
24OCT6:203SA3M	Sediment	Metals	379GLBR*146
25OCT9:194SA3M	Sediment	Metals	379GLBR*150
25OCT9:287SA3M	Sediment	Metals	379GLBR*155
25OCT9:338SA3M	Sediment	Metals	379GLBR*168
25OCT2:153SB1M	Sediment	Metals	379GLBR*177
25OCT2:334SB1M	Sediment	Metals	379GLBR*186
25OCT3:517SB1M	Sediment	Metals	379GLBR*192
25OCT3:558SB1M	Sediment	Metals	379GLBR*195
30OCT4:463SB2M	Sediment	Metals	379GLBR*207
31OCT9:007SB2M	Sediment	Metals	379GLBR*208
31OCT9:088SB2M	Sediment	Metals	379GLBR*211
31OCT9:274SB2M	Sediment	Metals	379GLBR*215
31OCT2:384SB3M	Sediment	Metals	379GLBR*245

31OCT10:453SB3M	Sediment	Metals	379GLBR*246
1NOV10:208SB3M	Sediment	Metals	379GLBR*265
1NOV10:157SB3M	Sediment	Metals	379GLBR*267
18NOV4:403SC1M	Sediment	Metals	379GLBR*285
19NOV8:484SC1M	Sediment	Metals	379GLBR*300
19NOV9:218SC1M	Sediment	Metals	379GLBR*305
19NOV9:327SC1M	Sediment	Metals	379GLBR*308
19NOV3:553SC2M	Sediment	Metals	379GLBR*316
19NOV4:414SC2M	Sediment	Metals	379GLBR*320
20NOV8:157SC2M	Sediment	Metals	379GLBR*336
20NOV1:553SC3M	Sediment	Metals	379GLBR*345
20NOV3:184SC3M	Sediment	Metals	379GLBR*353
20NOV3:327SC3M	Sediment	Metals	379GLBR*356
20NOV3:268SC3M	Sediment	Metals	379GLBR*359
21NOV2:433SD1M	Sediment	Metals	379GLBR*381
21NOV4:578SD1M	Sediment	Metals	379GLBR*383
21NOV5:087SD1M	Sediment	Metals	379GLBR*386
21NOV5:174SD1M	Sediment	Metals	379GLBR*389
21NOV5:194SD1M	Sediment	Metals	379GLBR*390
23OCT4:459LA2M	Oil	Metals	379GLBR*127†
23OCT5:2510LA2M	Water	Metals	379GLBR*131†
23OCT5:2010LA2M	Water	Metals	379GLBR*133*†
25OCT10:2010LA3M	Water	Metals	379GLBR*163†
25OCT11:0010LA3M	Water	Metals	379GLBR*170*
25OCT10:009LA3M	Oil	Metals	379GLBR*171†
25OCT3:1210LB1M	Water	Metals	379GLBR*188†
25OCT3:3010LB1M	Water	Metals	379GLBR*189*
30OCT4:216LA3M	Water	Metals	379GLBR*203
31OCT10:089LB2M	Oil	Metals	379GLBR*223
31OCT10:2010LB2M	Water	Metals	379GLBR*228†
31OCT10:3010LB2M	Water	Metals	379GLBR*229*†
31OCT3:5010LB3M	Water	Metals	379GLBR*251*
31OCT2:5710LB3M	Water	Metals	379GLBR*254†
31OCT3:069LB3M	Oil	Metals	379GLBR*255
1NOV11:006LB2M	Water	Metals	379GLBR*270
1NOV2:506LB1B3M	Water	Metals	379GLBR*274
6NOV12:003LM	Water	Metals	379GLBR*277
18NOV5:309LC1M	Oil	Metals	379GLBR*290†
18NOV6:1810LC1M	Water	Metals	379GLBR*292*
18NOV6:1810LC1M	Water	Metals	379GLBR*293
19NOV5:559LC2M	Oil	Metals	379GLBR*328†
19NOV5:2010LC2M	Water	Metals	379GLBR*333*
19NOV5:3510LC2M	Water	Metals	379GLBR*334
20NOV2:173LC3M	Water	Metals	379GLBR*362*
20NOV2:173LC3M	Water	Metals	379GLBR*363
20NOV2:439LC3M	Oil	Metals	379GLBR*368†
20NOV3:0310LC3M	Water	Metals	379GLBR*371
20NOV3:0710LC3M	Water	Metals	379GLBR*372*
21NOV3:3810LD1M	Water	Metals	379GLBR*399†
21NOV3:4510LD1M	Water	Metals	379GLBR*400*
21NOV4:359LD1M	Oil	Metals	379GLBR*405†

\*Samples were filtered at the time of collection therefore, results are for dissolved metals.

†Samples had a pH significantly greater than 2.

#### ARCHIVED SAMPLES

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Bartelle ID</u>
07OCT1:121SBM	Sediment	Metals	379GLBR*30
08OCT4:502SDM	Sediment	Metals	379GLBR*49-T2
08OCT4:472SDM	Sediment	Metals	379GLBR*49-T3
08OCT4:552SDM	Sediment	Metals	379GLBR*49-M1

08OCT4:522SDM	Sediment	Metals	379GLBR*49-M2
21OCT12:503SA1M	Sediment	Metals	379GLBR*81
21OCT5:484SA1M	Sediment	Metals	379GLBR*86
30OCT4:493SB2M	Sediment	Metals	379GLBR*207-T1
30OCT4:503SB2M	Sediment	Metals	379GLBR*207-T2
30OCT4:473SB2M	Sediment	Metals	379GLBR*207-M1
30OCT4:483SB2M	Sediment	Metals	379GLBR*207-M2
19NOV4:414SC2M	Sediment	Metals	379GLBR*320-T1
19NOV4:414SC2M	Sediment	Metals	379GLBR*320-T2
19NOV4:414SC2M	Sediment	Metals	379GLBR*320-M1
19NOV4:414SC2M	Sediment	Metals	379GLBR*320-M2
22NOV1:453SD2M	Sediment	Metals	379GLBR*413
22NOV3:184SD2M	Sediment	Metals	379GLBR*421
22NOV3:164SD2M	Sediment	Metals	379GLBR*422
22NOV3:348SD2M	Sediment	Metals	379GLBR*425
25NOV2:203SD3M	Sediment	Metals	379GLBR*441
25NOV4:004SD3M	Sediment	Metals	379GLBR*442
25NOV4:004SD3M	Sediment	Metals	379GLBR*448
21OCT6:2510LA1M	Water	Metals	379GLBR*96
21OCT6:459LA1M	Oil	Metals	379GLBR*102†
18NOV5:309LC1M	Oil	Metals	379GLBR*290-T1†
18NOV5:309LC1M	Oil	Metals	379GLBR*290-T2†
18NOV5:309LC1M	Oil	Metals	379GLBR*290-M1†
18NOV5:309LC1M	Oil	Metals	379GLBR*290-M2†
19NOV5:2010LC2M	Water	Metals	379GLBR*333-T2
19NOV5:2010LC2M	Water	Metals	379GLBR*333-M2
19NOV5:3510LC2M	Water	Metals	379GLBR*334-T1
19NOV5:3510LC2M	Water	Metals	379GLBR*334-T2
21NOV4:359LD1M	Oil	Metals	379GLBR*405-T1†
21NOV4:359LD1M	Oil	Metals	379GLBR*405-T2†
21NOV4:359LD1M	Oil	Metals	379GLBR*405-M1†
21NOV4:359LD1M	Oil	Metals	379GLBR*405-M2†
22NOV2:3210LD2M	Water	Metals	379GLBR*429†
22NOV2:4010LD2M	Water	Metals	379GLBR*430*
22NOV2:559LD2M	Oil	Metals	379GLBR*434†
25NOV4:0010LD3M	Water	Metals	379GLBR*455
25NOV4:009LD3M	Oil	Metals	379GLBR*457†
25NOV4:0010LD3M	Water	Metals	379GLBR*459

\*Samples were filtered at the time of collection, therefore results are for dissolved metals.

†Samples had a pH significantly greater than 2.

Some samples were not acidified at the time of collection to a pH  $\leq 2$ . Metals in water samples stored for any period of time at a pH  $> 2$  tend to adsorb onto the walls of the container which usually provide lower results. It became apparent in March, after all samples had been analyzed, that some samples were not acidified properly when mold was detected in those samples. We provided the acid normally required to acidify freshwater samples to a pH  $< 2$ . Therefore, either the acid was not added to the sample or the alkalinity was so high in the sample that the amount of acid provide was not sufficient to bring the pH down to 2. Those samples have been acidified since then and are available for re-analysis.

The water samples collected for metals analysis contained a high content of oil. As indicated by GLNPO, we digested the water including oil with hot nitric acid to obtain a total metals value. Values for samples flagged with an F represent total dissolved metals in the entire sample (water + oil).

The high standard for lead in water has a concentration of 1 mg/L and a milliabsorbance reading of 47. Some water samples analyzed for lead had a milliabsorbance reading slightly higher than 47 (48-55). The curve should still be linear at these milliabsorbance values, therefore would not affect the quality of

the data. One sample had a milll absorbance reading of 66 which was diluted and rerun by graphite furnace.

Deviations of the precision acceptance criteria for chromium were found on samples that had concentrations near the detection limit of 25 µg/g. Samples that had higher chromium concentrations were well within the precision acceptance criteria established in the QA plan.

Water samples for mercury were analyzed by the water method initially. As the analyst became aware of matrix problems, she digested and analyzed all the samples as sediments. The results on the first raw data sheet are for mercury by the water method. Those results were not reported. When applicable, duplicate analyses were averaged and the average was reported. Recoveries for one set of matrix spikes for mercury in water were outside the criteria. Recoveries were high (128 and 155%) due to very large dilution factors necessary to obtain results within the calibration curve, however this should not compromise the data.

## SOLIDS and TOC

Ninety-six sediment samples and twenty-two water samples were analyzed for solids content and total organic carbon. An aliquot of each sample was oven-dried at 105°±5°C according to Battelle SOP# MSL-M-3, then taken to 550°±10°C to allow volatilization according to Battelle SOP# MSL-M-2. A separate aliquot of each sample was freeze-dried according to Battelle SOP# MSL-M-3 and sent to Global Geochemistry Laboratory for total organic carbon analysis according to the LECO method for TOC in weight percent. Water samples were analyzed for total solids according to Battelle SOP# MSL-M-40 (based on Standard Methods, 2540B) and total suspended solids according to Battelle SOP# MSL-M-39 (based on Standard Methods, 2540D). With such a high amount of heavy particulates, it was difficult for the analyst to obtain a representative sample for total suspended solids and total solids. All solids samples were analyzed within the holding time of 7 days from collection. However, if the reproducibility was outside the acceptance criteria of ±20% the samples were re-analyzed, usually after the holding time had been exceeded. Separate samples collected at the sampling site were sent to Analytical Resources, Inc. for total organic carbon. There was a problem with high particulate concentrations in the samples. After some discussion with Eric Crecelius (program manager), they were instructed to allow the particulates to settle, then withdraw a portion of the liquid phase for analysis.

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
07OCT2:091SDS	Sediment	TS, TVS, TOC	379GLBR*34
07OCT1:521SDS	Sediment	TS, TVS, TOC	379GLBR*35
07OCT1:111SBS	Sediment	TS, TVS, TOC	379GLBR*36
07OCT1:201SCS	Sediment	TS, TVS, TOC	379GLBR*37
07OCT1:431SCS	Sediment	TS, TVS, TOC	379GLBR*38
07OCT12:411SAS	Sediment	TS, TVS, TOC	379GLBR*39
07OCT1:331SCS	Sediment	TS, TVS, TOC	379GLBR*40
07OCT1:021SBS	Sediment	TS, TVS, TOC	379GLBR*41
07OCT12:501SBS	Sediment	TS, TVS, TOC	379GLBR*42
07OCT2:051SDS	Sediment	TS, TVS, TOC	379GLBR*43
07OCT12:321SAS	Sediment	TS, TVS, TOC	379GLBR*44
07OCT12:121SAS	Sediment	TS, TVS, TOC	379GLBR*45
08OCT5:312SD2S	Sediment	TS, TVS, TOC	379GLBR*48
08OCT5:412SD1S	Sediment	TS, TVS, TOC	379GLBR*50
08OCT5:172SD3S	Sediment	TS, TVS, TOC	379GLBR*52
09OCT11:282SA1S	Sediment	TS, TVS, TOC	379GLBR*54
09OCT11:382SA2S	Sediment	TS, TVS, TOC	379GLBR*55
09OCT11:502SA3S	Sediment	TS, TVS, TOC	379GLBR*56
09OCT9:552SB1S	Sediment	TS, TVS, TOC	379GLBR*62
09OCT10:152SB2S	Sediment	TS, TVS, TOC	379GLBR*63
09OCT10:352SB3S	Sediment	TS, TVS, TOC	379GLBR*64

09OCT4:362SC2S	Sediment	TS, TVS, TOC	379GLBR*70
09OCT4:552SC1S	Sediment	TS, TVS, TOC	379GLBR*71
09OCT4:152SC3S	Sediment	TS, TVS, TOC	379GLBR*72
22OCT11:307SA1S	Sediment	TS, TVS, TOC	379GLBR*98
22OCT11:368SA1S	Sediment	TS, TVS, TOC	379GLBR*105
22OCT4:203SA2S	Sediment	TS, TVS, TOC	379GLBR*109
22OCT5:553SA2S	Sediment	TS, TVS, TOC	379GLBR*110
23OCT1:303SA2O	Sediment	TS, TVS, TOC	379GLBR*114
23OCT4:254SA2S	Sediment	TS, TVS, TOC	379GLBR*116
23OCT4:264SA2S	Sediment	TS, TVS, TOC	379GLBR*117
23OCT4:274SA2S	Sediment	TS, TVS, TOC	379GLBR*118
24OCT10:457SA2S	Sediment	TS, TVS, TOC	379GLBR*134
24OCT10:508SA2S	Sediment	TS, TVS, TOC	379GLBR*137
24OCT2:203SA3S	Sediment	TS, TVS, TOC	379GLBR*140
24OCT5:503SA3S	Sediment	TS, TVS, TOC	379GLBR*142
24OCT6:203SA3S	Sediment	TS, TVS, TOC	379GLBR*144
25OCT9:234SA3S	Sediment	TS, TVS, TOC	379GLBR*147
25OCT9:244SA3S	Sediment	TS, TVS, TOC	379GLBR*148
25OCT9:254SA3S	Sediment	TS, TVS, TOC	379GLBR*149
25OCT9:307SA3S	Sediment	TS, TVS, TOC	379GLBR*157
25OCT9:358SA3S	Sediment	TS, TVS, TOC	379GLBR*169
25OCT12:153SB1S	Sediment	TS, TVS, TOC	379GLBR*173
30OCT2:573SB2S	Sediment	TS, TVS, TOC	379GLBR*172
25OCT2:153SB1S	Sediment	TS, TVS, TOC	379GLBR*175
25OCT2:304SB1S	Sediment	TS, TVS, TOC	379GLBR*178
25OCT2:314SB1S	Sediment	TS, TVS, TOC	379GLBR*179
25OCT2:324SB1S	Sediment	TS, TVS, TOC	379GLBR*180
25OCT3:517SB1S	Sediment	TS, TVS, TOC	379GLBR*191
25OCT3:568SB1S	Sediment	TS, TVS, TOC	379GLBR*194
30OCT4:053SB2S	Sediment	TS, TVS, TOC	379GLBR*200
30OCT4:393SB2S	Sediment	TS, TVS, TOC	379GLBR*205
31OCT9:017SB2S	Sediment	TS, TVS, TOC	379GLBR*209
31OCT9:068SB2S	Sediment	TS, TVS, TOC	379GLBR*212
31OCT9:204SB2S	Sediment	TS, TVS, TOC	379GLBR*216
31OCT9:234SB2S	Sediment	TS, TVS, TOC	379GLBR*217
31OCT9:224SB2S	Sediment	TS, TVS, TOC	379GLBR*218
31OCT2:364SB3S	Sediment	TS, TVS, TOC	379GLBR*235
31OCT11:503SB3S	Sediment	TS, TVS, TOC	379GLBR*236
31OCT10:453SB3S	Sediment	TS, TVS, TOC	379GLBR*237
31OCT2:303SB3S	Sediment	TS, TVS, TOC	379GLBR*238
31OCT2:374SB3S	Sediment	TS, TVS, TOC	379GLBR*239
31OCT2:374SB3S	Sediment	TS, TVS, TOC	379GLBR*240
1NOV10:208SB3S	Sediment	TS, TVS, TOC	379GLBR*264
1NOV10:157SB3S	Sediment	TS, TVS, TOC	379GLBR*266
18NOV2:553SC1S	Sediment	TS, TVS, TOC	379GLBR*279
18NOV3:503SC1S	Sediment	TS, TVS, TOC	379GLBR*281
18NOV4:403SC1S	Sediment	TS, TVS, TOC	379GLBR*283
19NOV8:554SC1S	Sediment	TS, TVS, TOC	379GLBR*296
19NOV8:564SC1S	Sediment	TS, TVS, TOC	379GLBR*297
19NOV8:574SC1S	Sediment	TS, TVS, TOC	379GLBR*298
19NOV9:228SC1S	Sediment	TS, TVS, TOC	379GLBR*306
19NOV9:337SC1S	Sediment	TS, TVS, TOC	379GLBR*309
19NOV11:303SC2S	Sediment	TS, TVS, TOC	379GLBR*310
19NOV2:203SC2S	Sediment	TS, TVS, TOC	379GLBR*312
19NOV3:553SC2S	Sediment	TS, TVS, TOC	379GLBR*314
19NOV4:314SC2S	Sediment	TS, TVS, TOC	379GLBR*317
19NOV4:314SC2S	Sediment	TS, TVS, TOC	379GLBR*318
19NOV4:314SC2S	Sediment	TS, TVS, TOC	379GLBR*319
20NOV8:157SC2S	Sediment	TS, TVS, TOC	379GLBR*338
20NOV11:443SC3S	Sediment	TS, TVS, TOC	379GLBR*339
20NOV1:003SC3S	Sediment	TS, TVS, TOC	379GLBR*341

20NOV1:553SC3S	Sediment	TS, TVS, TOC	379GLBR*343
20NOV3:124SC3S	Sediment	TS, TVS, TOC	379GLBR*347
20NOV3:124SC3S	Sediment	TS, TVS, TOC	379GLBR*348
20NOV3:124SC3S	Sediment	TS, TVS, TOC	379GLBR*349
20NOV3:317SC3S	Sediment	TS, TVS, TOC	379GLBR*355
20NOV3:258SC3S	Sediment	TS, TVS, TOC	379GLBR*358
21NOV12:003SD1S	Sediment	TS, TVS, TOC	379GLBR*375
21NOV1:353SD1S	Sediment	TS, TVS, TOC	379GLBR*377
21NOV2:433SD1S	Sediment	TS, TVS, TOC	379GLBR*379
21NOV4:558SD1S	Sediment	TS, TVS, TOC	379GLBR*382
21NOV5:087SD1S	Sediment	TS, TVS, TOC	379GLBR*387
21NOV5:214SD1S	Sediment	TS, TVS, TOC	379GLBR*392
21NOV5:214SD1S	Sediment	TS, TVS, TOC	379GLBR*393
21NOV5:214SD1S	Sediment	TS, TVS, TOC	379GLBR*394
23OCT4:459LA2S	Oil	TS, TSS	379GLBR*126
23OCT5:2010LA2S	Water	TS, TSS	379GLBR*130
25OCT10:2210LA3S	Water	TS, TSS	379GLBR*158
25OCT10:2110LA3S	Water	TS, TSS	379GLBR*159
25OCT3:1110LB1S	Water	TS, TSS	379GLBR*197
30OCT4:166LA3S	Water	TS, TSS	379GLBR*202
31OCT10:059LB2S	Oil	TS, TSS	379GLBR*224
31OCT10:1910LB2S	Water	TS, TSS	379GLBR*230
31OCT2:5410LB3S	Water	TS, TSS	379GLBR*258
31OCT2:5610LB3S	Water	TS, TSS	379GLBR*259
31OCT3:049LB3S	Oil	TS, TSS	379GLBR*260
31OCT3:059LB3S	Oil	TS, TSS	379GLBR*261
6NOV12:003LS	Water	TS, TSS	379GLBR*276
18NOV5:409LC1S	Oil	TS, TSS	379GLBR*289
18NOV6:1010LC1S	Water	TS, TSS	379GLBR*291
19NOV5:539LC2S	Oil	TS, TSS	379GLBR*329
19NOV5:2410LC2S	Water	TS, TSS	379GLBR*332
20NOV2:163LC3S	Water	TS, TSS	379GLBR*364
20NOV2:409LC3S	Oil	TS, TSS	379GLBR*367
20NOV3:0010LC3S	Water	TS, TSS	379GLBR*373
21NOV3:3510LD1S	Water	TS, TSS	379GLBR*401
21NOV4:039LD1S	Oil	TS, TSS	379GLBR*403
3OCT4:459LA2O	Oil	TOC	379GLBR*125
23OCT5:2010LA2C	Water	TOC	379GLBR*129
25OCT10:2110LA3C	Water	TOC	379GLBR*162
25OCT10:009LA3C	Oil	TOC	379GLBR*165
25OCT3:1110LB1C	Water	TOC	379GLBR*190
30OCT4:206LA3C	Water	TOC	379GLBR*201
31OCT10:109LB2C	Oil	TOC	379GLBR*225
31OCT10:2410LB2C	Water	TOC	379GLBR*231
31OCT2:5810LB3C	Water	TOC	379GLBR*262
31OCT3:099LB3C	Oil	TOC	379GLBR*263
1NOV11:006LB2C	Water	TOC	379GLBR*271
1NOV2:506LB1B3C	Water	TOC	379GLBR*273
6NOV12:003LC	Water	TOC	379GLBR*278
18NOV6:0610LC1C	Water	TOC	379GLBR*294
18NOV5:459LC1C	Oil	TOC	379GLBR*295
19NOV5:579LC2C	Oil	TOC	379GLBR*330
19NOV5:4310LC2C	Water	TOC	379GLBR*335
20NOV2:183LC3C	Water	TOC	379GLBR*365
20NOV2:449LC3C	Oil	TOC	379GLBR*369
20NOV3:0210LC3C	Water	TOC	379GLBR*374
21NOV3:3710LD1C	Water	TOC	379GLBR*402
21NOV4:459LD1C	Oil	TOC	379GLBR*406

**BUFFALO PILOT PROJECT (CF# 379)  
GRAIN SIZE - TOTAL SOLIDS ANALYSIS**

MSL Code	Sponsor ID	% Total Solids	Predicted Dry Mass (g)	Actual Dry Mass (g)	Estimated Recovery
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**BIN A**

379GLBR-32	07OCT12:161SAG	60.05%	14.2368	15.2327	107.00%
379GLBR-61	09OCT12:002SAG	64.44%	13.5706	13.7666	101.44%
379GLBR-120	23OCT4:304SA2G	99.87%	10.4067	10.0779	96.84%
379GLBR-156	25OCT9:264SA3G	99.55%	9.2782	10.2376	110.34%

**BIN B**

379GLBR-31	07OCT12:511SBG	54.78%	14.4168	15.7037	108.93%
379GLBR-69	09OCT10:502SBG	59.75%	12.2001	13.2007	108.20%
379GLBR-184	25OCT2:324SB1G	98.94%	9.3495	10.1831	108.92%
379GLBR-219	31OCT9:274SB2G	100.09%	9.5281	10.1853	106.90%
379GLBR-241	31OCT2:364SB3G	100.01%	9.8506	10.5914	107.52%

**BIN C**

379GLBR-25	07OCT1:221SCG	65.29%	14.5604	15.0865	103.61%
379GLBR-77	09OCT5:082SCG	60.00%	11.9039	13.0577	109.69%
379GLBR-299	19NOV8:584SC1G	100.05%	10.3054	10.4093	101.01%
379GLBR-321	Rep 1 19NOV4:464SC2G	100.11%	9.8212	10.4951	106.86%
379GLBR-321	Rep 2 19NOV4:464SC2G	100.14%	9.8535	10.3475	105.01%
379GLBR-321	Rep 3 19NOV4:464SC2G	100.14%	9.9439	10.5172	105.77%
379GLBR-346	20NOV3:114SC3G	96.79%	9.4950	10.1702	107.11%

**BIN D**

379GLBR-33	07OCT1:561SDG	59.83%	13.2771	14.1625	106.67%
379GLBR-47	Rep 1 08OCT5:222SD2G	75.50%	14.6170	12.6762	86.72%
379GLBR-47	Rep 2 08OCT5:222SD2G	58.80%	11.3138	12.5171	110.64%
379GLBR-47	Rep 3 08OCT5:222SD2G	58.80%	11.4663	12.4534	108.61%
379GLBR-391	21NOV5:204SD1G	99.97%	9.2072	10.0154	108.78%
379GLBR-346 *	20NOV3:114SC3G	96.79%	9.4950	10.1720	107.11%
379GLBR-391 *	21NOV5:204SD1G	99.93%	10.2629	10.8617	105.83%

\* These dry samples were not disaggregated before analysis was done. They should be used only as a comparison for the rest of the dry samples which were disaggregated before analysis.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
GRAIN SIZE - TOTAL SOLIDS ANALYSIS**

		PERCENT OF TOTAL MASS								
MSL Code	Sponsor ID	>2.00 mm	2.00 mm	1.00 mm	0.500- 1.00 mm	0.25- 0.500 mm	0.125- 0.250 mm	0.0625- 0.125 mm	48.0- 62.5 um	31.2- 48.0 um
BIN A										
379GLBR-32	07OCT12:161SAG	0.06	0.11	0.15	0.53	2.67	10.29	3.73	6.38	
379GLBR-61	09OCT12:002SAG	0.40	0.05	0.20	1.12	3.83	12.19	3.60	5.99	
379GLBR-120	23OCT4:304SA2G	0.00	0.21	0.38	1.91	4.75	14.32	2.58	11.23	
379GLBR-156	25OCT9:264SA3G	0.00	0.38	0.91	1.28	3.85	13.25	6.90	8.38	
BIN B										
379GLBR-31	07OCT12:511SBG	0.08	0.06	0.22	0.66	1.78	7.46	4.79	3.23	
379GLBR-69	09OCT10:502SBG	0.13	0.14	0.27	1.48	4.90	13.75	3.30	7.55	
379GLBR-184	25OCT2:324SB1G	0.04	0.16	0.36	1.86	9.10	16.40	2.51	11.51	
379GLBR-219	31OCT9:274SB2G	0.00	0.24	0.33	2.06	7.65	17.86	9.90	6.99	
379GLBR-241	31OCT2:364SB3G	0.04	0.19	0.59	3.18	6.03	16.82	1.40	8.84	
BIN C										
379GLBR-25	07OCT1:221SCG	0.10	0.13	0.23	1.45	15.60	15.58	1.22	7.87	
379GLBR-77	09OCT5:082SCG	0.08	0.14	0.20	1.09	4.86	13.74	4.56	5.76	
379GLBR-299	19NOV8:584SC1G	0.00	0.31	0.42	2.17	8.53	18.63	7.99	7.38	
379GLBR-321	Rep 1 19NOV4:464SC2G	0.00	0.39	0.34	3.24	13.37	14.17	5.49	8.65	
379GLBR-321	Rep 2 19NOV4:464SC2G	0.11	0.07	0.48	3.56	10.75	17.68	8.39	7.19	
379GLBR-321	Rep 3 19NOV4:464SC2G	0.00	0.11	0.45	3.90	10.24	17.52	6.28	9.28	
379GLBR-346	20NOV3:114SC3G	0.27	0.66	0.98	2.52	8.44	17.27	6.88	7.98	
BIN D										
379GLBR-33	07OCT1:561SDG	0.10	0.05	0.14	0.73	4.33	13.79	3.39	8.08	
379GLBR-47	Rep 1 08OCT5:222SD2G	0.02	0.09	0.32	1.13	3.61	12.76	4.23	7.57	
379GLBR-47	Rep 2 08OCT5:222SD2G	0.09	0.14	0.26	1.17	3.88	12.50	2.05	7.22	
379GLBR-47	Rep 3 08OCT5:222SD2G	0.82	0.16	0.25	1.10	3.54	12.58	2.18	8.87	
379GLBR-391	21NOV5:204SD1G	0.00	0.14	0.66	4.40	8.32	16.38	2.16	8.43	
379GLBR-346	20NOV3:114SC3G	30.59	15.06	9.86	4.84	5.79	7.78	1.24	3.14	
379GLBR-391	21NOV5:204SD1G	33.61	17.82	11.90	5.37	3.97	6.34	2.03	1.84	

NOTE: All results are in percent.  
 \* These dry samples were not disaggregated before analysis was done.  
 They should be used only as a comparison for the rest of the  
 dry samples which were disaggregated before analysis.



**BUFFALO RIVER PILOT PROJECT (CF#379)  
GRAIN SIZE - TOTAL SOLIDS ANALYSIS**

		PERCENT OF TOTAL MASS											
MSL Code	Sponsor ID	23.0- 31.2 um	15.6- 23.0 um	7.8- 15.6 um	3.9- 7.8 um	1.9- 3.9 um	0.976- 1.9 um	0.488- 0.976 um	<0.488 um	Salt	Blank		
BIN A													
379GLBR-32	07OCT12:161SAG	10.50	10.61	20.06	10.48	7.59	5.17	3.12	8.38	0.0158			
379GLBR-61	09OCT12:002SAG	8.08	10.87	17.84	9.24	6.57	4.68	3.31	12.03	0.0072			
379GLBR-120	23OCT4:304SA2G	11.35	11.35	19.85	8.57	6.03	3.81	2.66	0.99	0.0009			
379GLBR-156	25OCT9:264SA3G	10.79	11.65	21.89	2.96	4.75	6.66	0.43	5.92	0.0005			
BIN B													
379GLBR-31	07OCT12:511SBG	10.49	10.55	21.17	12.00	8.35	5.09	3.72	10.34	0.0111			
379GLBR-69	09OCT10:502SBG	8.09	9.48	17.21	8.48	6.39	4.85	3.21	10.76	0.0109			
379GLBR-184	25OCT2:324SB1G	10.29	8.60	14.02	8.68	5.66	4.24	0.86	5.70	0.0004			
379GLBR-219	31OCT9:274SB2G	10.17	9.86	15.16	8.25	5.07	3.50	1.96	1.02	0.0003			
379GLBR-241	31OCT2:364SB3G	9.44	8.76	23.79	8.57	5.36	0.04	2.04	4.91	0.0005			
BIN C													
379GLBR-25	07OCT1:221SCG	6.95	8.46	12.20	9.68	5.30	3.37	2.81	9.07	0.0119			
379GLBR-77	09OCT5:082SCG	10.14	9.59	16.76	8.73	5.67	4.07	3.37	11.24	0.0105			
379GLBR-299	19NOV8:584SC1G	7.92	7.99	21.17	2.96	7.72	0.77	3.34	2.69	0.0004			
379GLBR-321	Rep 1 19NOV4:464SC2G	8.16	7.47	15.51	7.55	9.68	3.28	0.95	1.75	0.0006			
379GLBR-321	Rep 2 19NOV4:464SC2G	7.38	7.50	14.61	6.84	5.03	8.12	0.89	1.39	0.0011			
379GLBR-321	Rep 3 19NOV4:464SC2G	8.10	7.53	13.92	6.47	5.25	8.48	0.65	1.83	0.0007			
379GLBR-346	20NOV3:114SC3G	8.14	7.35	16.91	7.91	5.03	3.85	4.88	0.90	0.0003			
BIN D													
379GLBR-33	07OCT1:561SDG	8.08	10.20	18.08	9.46	6.44	4.80	3.50	8.84	0.0156			
379GLBR-47	Rep 1 08OCT5:222SD2G	7.76	10.67	16.82	9.09	6.41	4.29	3.41	11.83	0.0098			
379GLBR-47	Rep 2 08OCT5:222SD2G	9.65	9.81	17.70	8.66	6.20	4.44	3.32	12.91	0.0087			
379GLBR-47	Rep 3 08OCT5:222SD2G	8.42	11.15	17.18	9.06	6.33	4.53	3.31	10.54	0.0157			
379GLBR-391	21NOV5:204SD1G	8.55	10.78	20.45	8.07	4.75	3.99	2.12	0.80	0.0006			
379GLBR-346 *	20NOV3:114SC3G	1.98	3.29	6.78	2.90	1.34	0.99	0.14	4.28	0.0222			
379GLBR-391 *	21NOV5:204SD1G	2.25	2.32	5.08	1.58	0.88	0.48	0.37	4.16	0.0191			

NOTE: All results are in percent.  
 \* These dry samples were not disaggregated before analysis was done.  
 They should be used only as a comparison for the rest of the  
 dry samples which were disaggregated before analysis.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Cr (ug/g) XRF	Cu (ug/g) XRF	Pb (ug/g) XRF
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**BIN A**

379GLBR- 8	07OCT12:301SAL	77	39.8	46.0
379GLBR- 27	07OCT12:191SAM	73	46.2	72.2
379GLBR- 60	09OCT12:002SAM	60	45.8	52.4
379GLBR- 103	22OCT11:287SA1M	110	55.3	65.6
379GLBR- 106	22OCT11:358SA1M	155	55.4	56.6
379GLBR- 115	23OCT1:303SA2M	47	41.1	53.7
379GLBR- 119	23OCT4:314SA2M	70	38.2	52.1
379GLBR- 135	24OCT10:457SA2M	137	51.3	58.9
379GLBR- 138	24OCT10:508SA2M	84	42.3	48.8
379GLBR- 146	24OCT6:203SA3M	68	40.6	52.4
379GLBR- 150	25OCT9:194SA3M	61	41.3	52.7
379GLBR- 154	25OCT9:104SA3M	66	42.7	54.9
379GLBR- 155	25OCT9:287SA3M	148	47.1	56.7
379GLBR- 168	25OCT9:338SA3M	88	43.1	48.0
379GLBR- 462	4DEC12:055SA2M	100	41.6	50.5

**BIN B**

379GLBR- 29	07OCT12:541SBM	63	41.0	49.3
379GLBR- 68	09OCT10:502SBM	51	43.0	58.2
379GLBR- 177	25OCT2:153SB1M	47	37.6	64.3
379GLBR- 186	25OCT2:334SB1M	56	45.7	65.7
379GLBR- 192	25OCT3:517SB1M	129	48.2	65.1
379GLBR- 195	25OCT3:558SB1M	121	46.2	58.4
379GLBR- 207 Rep 1	30OCT4:463SB2M	46	44.9	65.7
379GLBR- 207 Rep 2	30OCT4:463SB2M	70	41.1	56.4
379GLBR- 207 Rep 3	30OCT4:463SB2M	57	43.0	62.9
379GLBR- 208	31OCT9:007SB2M	106	46.8	67.0
379GLBR- 211	31OCT9:088SB2M	77	47.1	64.5
379GLBR- 215	31OCT9:274SB2M	44	45.6	67.0
379GLBR- 245	31OCT2:384SB3M	58	44.1	61.1
379GLBR- 246	31OCT10:453SB3M	55	43.2	60.6
379GLBR- 265	1NOV10:208SB3M	117	49.5	61.3
379GLBR- 267	1NOV10:157SB3M	130	47.8	62.3

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Cr (ug/g) XRF	Cu (ug/g) XRF	Pb (ug/g) XRF
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**BIN C**

379GLBR- 26	07OCT1:261SCM	80	56.8	88.8
379GLBR- 76	09OCT5:052SCM	71	47.8	62.3
379GLBR- 285	18NOV4:403SC1M	73	45.3	68.4
379GLBR- 300	19NOV8:484SC1M	67	58.0	70.6
379GLBR- 305	19NOV9:218SC1M	129	62.1	59.7
379GLBR- 308	19NOV9:327SC1M	136	54.8	83.8
379GLBR- 316	19NOV3:553SC2M	59	48.2	71.9
379GLBR- 320 Rep 1	19NOV4:414SC2M	75	47.6	68.6
379GLBR- 320 Rep 2	19NOV4:414SC2M	63	51.4	67.4
379GLBR- 320 Rep 3	19NOV4:414SC2M	37	47.4	65.5
379GLBR- 336	20NOV8:157SC2M	129	56.1	70.7
379GLBR- 345	20NOV1:553SC3M	69	47.2	56.3
379GLBR- 353	20NOV3:184SC3M	62	45.4	58.3
379GLBR- 356	20NOV3:327SC3M	140	62.4	70.4
379GLBR- 359	20NOV3:268SC3M	140	52.5	61.7

**BIN D**

379GLBR- 28	07OCT2:001SDM	74	37.7	49.7
379GLBR- 49 Rep 1	08OCT4:572SDM	49	44.5	65.0
379GLBR- 49 Rep 2	08OCT4:572SDM	56	42.8	60.3
379GLBR- 49 Rep 3	08OCT4:572SDM	76	47.1	61.7
379GLBR- 381	21NOV2:43ESD1M	55	44.8	73.5
379GLBR- 383	21NOV4:578SD1M	118	53.7	71.8
379GLBR- 386	21NOV5:087SD1M	112	51.3	63.6
379GLBR- 390	21NOV5:194SD1M	62	47.6	64.7

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Cr (ug/g) XFF	Cu (ug/g) XFF	Pb (ug/g) XFF
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**STANDARD REFERENCE MATERIAL**

SRM 1646	Rep 1	61	22.1	26.9
SRM 1646	Rep 2	78	19.7	28.3
SRM 1646	Rep 3	78	23.2	29.1
SRM1646	Rep 4	72	20.9	29.3
	<b>certified</b>	76	18	28.2
	<b>value</b>	±3	±3	±1.8
SRM 2704	Rep 1	140	91.6	156.5
SRM 2704	Rep 2	134	97.5	156.2
SRM 2704	Rep 3	139	92.9	155.1
	<b>certified</b>	135	98.6	161.0
	<b>value</b>	±5	±5.0	±17.0

**REPLICATE ANALYSIS**

379GLBR- 207	Rep 1	30OCT4:463SB2M	46	44.9	65.7
379GLBR- 207	Rep 2	30OCT4:463SB2M	70	41.1	56.4
379GLBR- 207	Rep 3	30OCT4:463SB2M	57	43	62.9
		<b>RSD %</b>	<b>21%</b>	<b>4%</b>	<b>8%</b>
379GLBR- 320	Rep 1	19NOV4:424SC2M	75	47.6	68.6
379GLBR- 320	Rep 2	19NOV4:424SC2M	63	51.4	67.4
379GLBR- 320	Rep 3	19NOV4:424SC2M	37	47.4	65.5
		<b>RSD %</b>	<b>33%</b>	<b>5%</b>	<b>2%</b>
379GLBR- 49	Rep 1	08OCT4:572SDM	49	44.5	65
379GLBR- 49	Rep 2	08OCT4:572SDM	56	42.8	60.3
379GLBR- 49	Rep 3	08OCT4:572SDM	76	47.1	61.7
		<b>RSD %</b>	<b>23%</b>	<b>5%</b>	<b>4%</b>

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Hg (ug/g) CVAA
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**BIN A**

379GLBR- 8	07OCT12:301SAL	0.112
379GLBR- 27	07OCT12:191SAM	0.183
379GLBR- 60	09OCT12:002SAM	0.192
379GLBR- 103	22OCT11:287SA1M	0.183
379GLBR- 106	22OCT11:358SA1M	0.089
379GLBR- 115	23OCT1:303SA2M	0.171
379GLBR- 119	23OCT4:314SA2M	0.002
379GLBR- 135	24OCT10:457SA2M	0.146
379GLBR- 138	24OCT10:508SA2M	0.023
379GLBR- 146	24OCT6:203SA3M	0.175
379GLBR- 150	25OCT9:194SA3M	0.045
379GLBR- 154	25OCT9:L104SA3M	0.027
379GLBR- 155	25OCT9:287SA3M	0.120
379GLBR- 168	25OCT9:338SA3M	0.036
379GLBR- 462	4DEC12:055SA2M	0.017

**BIN B**

379GLBR- 29	07OCT12:541SBM	0.170
379GLBR- 68	09OCT10:502SBM	0.208
379GLBR- 177	25OCT2:153SB1M	0.203
379GLBR- 186	25OCT2:334SB1M	0.111
379GLBR- 192	25OCT3:517SB1M	0.132
379GLBR- 195	25OCT3:558SB1M	0.121
379GLBR- 207 Rep 1	30OCT4:463SB2M	0.190
379GLBR- 207 Rep 2	30OCT4:463SB2M	0.205
379GLBR- 207 Rep 3	30OCT4:463SB2M	0.203
379GLBR- 208	31OCT9:007SB2M	0.102
379GLBR- 211	31OCT9:088SB2M	0.023
379GLBR- 215	31OCT9:274SB2M	<0.0003
379GLBR- 245	31OCT2:384SB3M	0.032
379GLBR- 246	31OCT10:453SB3M	0.173
379GLBR- 265	1NOV10:208SB3M	0.084
379GLBR- 267	1NOV10:157SB3M	0.175

**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Hg (ug/g) CVAA
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**BIN C**

379GLBR- 23	07OCT1:261SCM	0.335
379GLBR- 76	09OCT5:052SCM	0.198
379GLBR- 285	18NOV4:403SC1M	0.202
379GLBR- 300	19NOV8:484SC1M	0.065
379GLBR- 305	19NOV9:218SC1M	0.300
379GLBR- 308	19NOV9:327SC1M	0.323
379GLBR- 316	19NOV3:553SC2M	0.213
379GLBR- 320 Rep 1	19NOV4:414SC2M	0.046
379GLBR- 320 Rep 2	19NOV4:414SC2M	0.054
379GLBR- 320 Rep 3	19NOV4:414SC2M	0.052
379GLBR- 336	20NOV8:157SC2M	0.228
379GLBR- 345	20NOV1:553SC3M	0.175
379GLBR- 353	20NOV3:184SC3M	0.151
379GLBR- 356	20NOV3:327SC3M	0.487
379GLBR- 359	20NOV3:268SC3M	0.060

**BIN D**

379GLBR- 28	07OCT2:001SDM	0.110
379GLBR- 49 Rep 1	08OCT4:572SDM	0.214
379GLBR- 49 Rep 2	08OCT4:572SDM	0.207
379GLBR- 49 Rep 3	08OCT4:572SDM	0.233
379GLBR- 381	21NOV2:433SD1M	0.187
379GLBR- 383	21NOV4:578SD1M	0.034
379GLBR- 386	21NOV5:087SD1M	0.209
379GLBR- 390	21NOV5:194SD1M	0.014

Blank	Rep 1	<0.0003
Blank	Rep 2	<0.0003
Blank	Rep 3	<0.0003
Blank	Rep 4	<0.0003
Blank	Rep 5	<0.0003

**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Hg (ug/g) CVAA
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**STANDARD REFERENCE MATERIAL**

SRM 1646	Rep 1	0.072
SRM 1646	Rep 2	0.076
SRM 1646	Rep 3	0.070
SRM 1646	Rep 4	0.066
	<b>certified value</b>	<b>0.063 ±0.012</b>

SRM 2704	Rep 1	1.434
SRM 2704	Rep 2	1.439
SRM 2704	Rep 3	1.450
	<b>certified value</b>	<b>1.44 ±0.07</b>

**MATRIX SPIKE RESULTS**

Amount Spiked		0.500
379GLBR-8	07OCT12:301SAL	0.112
379GLBR-8 + Spike		0.647
Amount Recovered		0.535
Percent Recovery		107%

Amount Spiked		0.500
379GLBR-8	DUPLICATE	0.112
379GLBR-8 + Spike		0.667
Amount Recovered		0.555
Percent Recovery		111%

Amount Spiked		0.500
379GLBR-49	08OCT4:572SDM	0.214
379GLBR-49 + Spike		0.649
Amount Recovered		0.435
Percent Recovery		87%

Amount Spiked		0.500
379GLBR-49	DUPLICATE	0.207
379GLBR-49 + Spike		0.639
Amount Recovered		0.432
Percent Recovery		86%

**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Hg (ug/g) CVAA
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**MATRIX SPIKE RESULTS**

Amount Spiked		0.500
379GLBR-207	30OCT4:463SB2M	0.190
379GLBR-207+ Spike		0.703
Amount Recovered		0.513
Percent Recovery		103%

Amount Spiked		0.500
379GLBR-207 DUPLICATE		0.205
379GLBR-207+ Spike		0.659
Amount Recovered		0.454
Percent Recovery		91%

Amount Spiked		0.500
379GLBR-320	19NOV4:424SC2M	0.046
379GLBR-320+ Spike		0.501
Amount Recovered		0.455
Percent Recovery		91%

Amount Spiked		0.500
379GLBR-320 DUPLICATE		0.054
379GLBR-320+ Spike		0.516
Amount Recovered		0.462
Percent Recovery		92%



**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Hg (ug/g) CVAA
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**REPLICATE ANALYSIS**

379GLBR- 207 Rep 1	30OCT4:463SB2M	0.190
379GLBR- 207 Rep 2	30OCT4:463SB2M	0.205
379GLBR- 207 Rep 3	30OCT4:463SB2M	0.203
	<b>RSD %</b>	<b>4%</b>
379GLBR- 320 Rep 1	19NOV4:424SC2M	0.046
379GLBR- 320 Rep 2	19NOV4:424SC2M	0.054
379GLBR- 320 Rep 3	19NOV4:424SC2M	0.052
	<b>RSD %</b>	<b>8%</b>
379GLBR- 49 Rep 1	08OCT4:572SDM	0.214
379GLBR- 49 Rep 2	08OCT4:572SDM	0.207
379GLBR- 49 Rep 3	08OCT4:572SDM	0.233
	<b>RSD %</b>	<b>6%</b>

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN WATER SAMPLES**

MSL Code	Sponsor ID	Cr ug/L	Cu ug/L	Pb ug/L
MDL - Flame AA		40	50	100
MDL - Graphite Furnace AA		0.22	0.72	1.03

**BIN A**

379GLBR- 127	23OCT4:459LA2M	13130	25590	12600	
379GLBR- 131	23OCT5:2510LA2M	1080	1350	1040	
379GLBR- 133 REP 1	23OCT5:2010LA2M	4.15 *	830	2.97 *	F
379GLBR- 133 REP 2	23OCT5:2010LA2M	4.40 *	830	2.97 *	F
379GLBR- 133 REP 3	23OCT5:2010LA2M	4.15 *	820	3.56 *	F
379GLBR- 163	25OCT10:2010LA3M	600	570	230	
379GLBR- 170	25OCT11:0010LA3M	2.89 *	410	1.19 *	F
379GLBR- 171	25OCT10:009LA3M	14200	7100	7100	
379GLBR- 203	30OCT4:216LA3M	1.13 *	4.17 *	1.19 *	

**BIN B**

379GLBR- 188	25OCT3:1210LB1M	1870	840	900	
379GLBR- 189	25OCT3:3010LB1M	2.52 *	340	3.56 *	F
379GLBR- 223	31OCT10:089LB2M	22.4 *	2800	32.6 *	
379GLBR- 228	31OCT10:2010LB2M	200	560	154 *	
379GLBR- 229 REP 1	31OCT10:3010LB2M	1.76 *	450	1.78 *	F
379GLBR- 229 REP 2	31OCT10:3010LB2M	1.76 *	450	2.37 *	F
379GLBR- 229 REP 3	31OCT10:3010LB2M	1.76 *	450	2.37 *	F
379GLBR- 251	31OCT3:5010LB3M	1.76 *	330	25.5 *	F
379GLBR- 254	31OCT2:5710LB3M	270	520	200	
379GLBR- 255	31OCT3:069LB3M	120	1830	140	
379GLBR- 270	1NOV11:006LB2M	2.39 *	5.83 *	1.19 *	
379GLBR- 274	1NOV2:506LB1B3M	1.51 *	14.2 *	1.19 *	

**BIN C**

379GLBR- 290	18NOV5:309LC1M	830	3660	810	
379GLBR- 292	18NOV6:1810LC1M	2.77 *	720	1.78 *	F
379GLBR- 293	18NOV6:1810LC1M	230	1410	330	
379GLBR- 328	19NOV5:559LC2M	410	2220	410	
379GLBR- 333 REP 1	19NOV5:2010LC2M	1.64 *	120	13.1 *	F
379GLBR- 333 REP 2	19NOV5:2010LC2M	1.64 *	110	14.2 *	F
379GLBR- 333 REP 3	19NOV5:2010LC2M	2.14 *	120	14.8 *	F
379GLBR- 334	19NOV5:3510LC2M	160	260	180	
379GLBR- 362	20NOV2:173LC3M	1.51 *	5.00 *	2.37 *	F
379GLBR- 363	20NOV2:173LC3M	1.51 *	7.91 *	4.15 *	

NOTE: Sample values are not blank-corrected.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN WATER SAMPLES**

MSL Code	Sponsor ID	Cr ug/L	Cu ug/L	Pb ug/L
MDL - Flame AA		40	50	100
MDL - Graphite Furnace AA		0.22	0.72	1.03

**BIN C**

379GLBR- 368	20NOV2:435LC3M	1140	1010	19.6 *	
379GLBR- 371	20NOV3:0310LC3M	170	190	160	
379GLBR- 372	20NOV3:0710LC3M	1.01 *	70	17.8 *	F

**BIN D**

379GLBR- 399	21NOV3:3810LD1M	340	860	250	
379GLBR- 400	21NOV3:4510LD1M	1.64 *	755 *	8.90 *	F
379GLBR- 405 REP 1	21NOV4:359LD1M	800	1950	580	
379GLBR- 405 REP 2	21NOV4:359LD1M	620	1950	560	
379GLBR- 405 REP 3	21NOV4:359LD1M	750	1950	560	
379GLBR- 429	22NOV2:3210LD2M	530	950	440	
379GLBR- 430	22NOV2:4010LD2M	2.52 *	630	9.49 *	F
379GLBR- 434	22NOV2:559LD2M	840	1310	540	
379GLBR- 457	25NOV4:009LD3M	790	3000	1620 *	

**DILUTION WATER**

379GLBR- 277	6NOV12:003LM	2.26 *	17.9 *	2.37 *
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Blank	Rep 1	(Flame AA)	40 U	50 U	100 U
Blank	Rep 2		40 U	50 U	100 U
Blank	Rep 3		40 U	50 U	100 U
Blank	Rep 4		40 U	50 U	100 U
Blank	Rep 1	(Graphite Furnace AA)	1.13 *	4.17 *	1.03 U*
Blank	Rep 2		6.67 *	4.17 *	1.03 U*
Blank	Rep 3		1.01 *	3.33 *	1.03 U*
Blank	Rep 4		1.13 *	4.58 *	1.03 U*

NOTE: Sample values are not blank-corrected.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN WATER SAMPLES**

MSL Code	Sponsor ID	Cr ug/L	Cu ug/L	Pb ug/L
MDL - Flame AA		40	50	100
MDL - Graphite Furnace AA		0.22	0.72	1.03

**STANDARD REFERENCE MATERIAL**

SRM 1643c	Rep 1	21.5 *	29.2 *	35.6 *
SRM 1643c	Rep 2	21.5 *	26.7 *	36.8 *
SRM 1643c	Rep 3	19.1 *	28.7 *	36.8 *
SRM 1643c	Rep 4	20.4 *	27.1 *	33.8 *
	<b>certified</b>	19.0	22.3	35.3
	<b>value</b>	±0.6	±2.8	±0.9

NOTE: SRM values are not blank-corrected.

**MATRIX SPIKE RESULTS - FLAME AA**

Amount Spiked	1000	1000	1000
379GLBR- 203	40 U	50 U	100 U
379GLBR- 203 + Spike	940	1030	1080
Amount Recovered	940	1030	1080
Percent Recovered	94%	103%	108%

Amount Spiked	1000	1000	1000
379GLBR- 203	40 U	50	100 U
379GLBR- 203 + Spike DUPLICATE	890	1050	1060
Amount Recovered	890	1050	1060
Percent Recovered	89%	105%	106%

Amount Spiked	1000	1000	1000
379GLBR- 274	40 U	50 U	100 U
379GLBR- 274 + Spike	890	990	1010
Amount Recovered	890	990	1010
Percent Recovered	89%	99%	101%

Amount Spiked	1000	1000	1000
379GLBR- 274	40 U	50 U	100 U
379GLBR- 274 + Spike DUPLICATE	1020	1140	1110
Amount Recovered	1020	1140	1110
Percent Recovered	102%	114%	111%

NOTE: Spike data run by Flame AA is not blank-corrected.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN WATER SAMPLES**

MSL Code	Sponsor ID	Cr ug/L	Cu ug/L	Pb ug/L
MDL - Flame AA		40	50	100
MDL - Graphite Furnace AA		0.22	0.72	1.03
Amount Spiked		1000	1000	1000
379GLBR- 362		40 U	50 U	100 U
379GLBR- 362 + Spike		1030	1020	1000
Amount Recovered		1030	1020	1000
Percent Recovered		103%	102%	100%
Amount Spiked		1000	1000	1000
379GLBR- 362		40 U	50 U	100 U
379GLBR- 362 + Spike DUPLICATE		1040	1040	980
Amount Recovered		1040	1040	980
Percent Recovered		104%	104%	98%
Amount Spiked		1000	1000	1000
379GLBR- 400		40 U	330	100 U
379GLBR- 400 + Spike		1040	1360	1020
Amount Recovered		1040	1030	1020
Percent Recovered		104%	103%	102%
Amount Spiked		1000	1000	1000
379GLBR- 400		40 U	330	100 U
379GLBR- 400 + Spike DUPLICATE		1040	1370	980
Amount Recovered		1040	1040	980
Percent Recovered		104%	104%	98%

NOTE: Spike data run by Flame AA is not blank-corrected

**MATRIX SPIKE RESULTS - GRAPHITE FURNACE AA**

Amount Spiked	1000	1000	1000
379GLBR- 203	0.22 U	0.72 U	1.19
379GLBR- 203 + Spike	1091	1110	919
Amount Recovered	1091	1110	917
Percent Recovered	109%	111%	92%
Amount Spiked	1000	1000	1000
379GLBR- 203	0.22 U	0.72 U	1.19
379GLBR- 203 + Spike DUPLICATE	1062	977	912
Amount Recovered	1062	977	911
Percent Recovered	106%	98%	91%

NOTE: Spike data run by Graphite Furnace AA is blank-corrected.

**BUFFALO RIVER PILOT PROJECT (CF #379)**  
**METALS IN WATER SAMPLES**

MSL Code	Sponsor ID	Cr ug/L	Cu ug/L	Pb ug/L
MDL - Flame AA		40	50	100
MDL - Graphite Furnace AA		0.22	0.72	1.03
Amount Spiked		1000	1000	1000
379GLBR- 274		0.38	10.0	1.19
379GLBR- 274 + Spike		1120	933	1057
Amount Recovered		1120	923	1055
Percent Recovered		112%	92%	106%
Amount Spiked		1000	1000	1000
379GLBR- 274		0.38	10.0	1.19
379GLBR- 274 + Spike DUPLICATE		1152	1066	1057
Amount Recovered		1152	1056	1055
Percent Recovered		115%	106%	106%
Amount Spiked		1000	1000	1000
379GLBR- 362		0.38	0.83	2.37
379GLBR- 362 + Spike		1052	1155	977
Amount Recovered		1052	1154	974
Percent Recovered		105%	115%	97%
Amount Spiked		1000	1000	1000
379GLBR- 362		0.38	0.83	2.37
379GLBR- 362 + Spike DUPLICATE		1023	1066	957
Amount Recovered		1023	1065	955
Percent Recovered		102%	107%	95%
Amount Spiked		1000	1000	1000
379GLBR- 400		0.50	577	8.90
379GLBR- 400 + Spike		1101	1421	983
Amount Recovered		1100	844	974
Percent Recovered		110%	84%	97%
Amount Spiked		1000	1000	1000
379GLBR- 400		0.50	577	8.90
379GLBR- 400 + Spike DUPLICATE		1110	1643	964
Amount Recovered		1110	1066	955
Percent Recovered		111%	107%	95%

NOTE: Spike data run by Graphite Furnace AA is blank-corrected.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN WATER SAMPLES**

MSL Code	Sponsor ID	Cr ug/L	Cu ug/L	Pb ug/L
MDL - Flame AA		40	50	100
MDL - Graphite Furnace AA		0.22	0.72	1.03

**REPLICATE ANALYSIS**

379GLBR- 133 REP 1	23OCT5:2010LA2M	4.15 *	0.83	2.97 *	F
379GLBR- 133 REP 2	23OCT5:2010LA2M	4.40 *	0.83	2.97 *	F
379GLBR- 133 REP 3	23OCT5:2010LA2M	4.15 *	0.82	3.56 *	F
	RSD %	3%	1%	11%	
379GLBR- 229 REP 1	31OCT10:3010LB2M	1.76 *	0.45	1.78 *	F
379GLBR- 229 REP 2	31OCT10:3010LB2M	1.76 *	0.45	2.37 *	F
379GLBR- 229 REP 3	31OCT10:3010LB2M	1.76 *	0.45	2.37 *	F
	RSD %	0%	0%	16%	
379GLBR- 333 REP 1	19NOV5:2010LC2M	1.64 *	0.12	13.1 *	F
379GLBR- 333 REP 2	19NOV5:2010LC2M	1.64 *	0.11	14.2 *	F
379GLBR- 333 REP 3	19NOV5:2010LC2M	2.14 *	0.12	14.8 *	F
	RSD %	16%	5%	6%	
379GLBR- 405 REP 1	21NOV4:359LD1M	800	1950	580	
379GLBR- 405 REP 2	21NOV4:359LD1M	620	1950	560	
379GLBR- 405 REP 3	21NOV4:359LD1M	750	1950	560	
	RSD %	13%	0%	2%	

\* = Analyzed by Graphite Furnace AA

F = Samples were filtered prior to analysis.

U = Below detection limits

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN WATER SAMPLES**

MSL Code	Sponsor ID	Hg (ug/L) CVAA
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**BIN A**

379GLBR- 127	23OCT4:459LA2M	64.07
379GLBR- 131	23OCT5:2510LA2M	3.98
379GLBR- 133	23OCT5:2010LA2M	0.96
379GLBR- 163	25OCT10:2010LA3M	7.38
379GLBR- 170	25OCT11:0010LA3M	0.43
379GLBR- 171	25OCT10:009LA3M	3.27
379GLBR- 203	30OCT4:216LA3M	0.001

**BIN B**

379GLBR- 188	25OCT3:1210LB1M	1.28
379GLBR- 189	25OCT3:3010LB1M	0.52
379GLBR- 223	31OCT10:089LB2M	5.28
379GLBR- 228	31OCT10:2010LB2M	1.83
379GLBR- 229	31OCT10:3010LB2M	1.11
379GLBR- 251	31OCT3:5010LB3M	5.39
379GLBR- 254	31OCT2:5710LB3M	2.37
379GLBR- 255	31OCT3:069LB3M	2.40
379GLBR- 270	1NOV11:006LB2M	< 0.005
379GLBR- 274	1NOV2:506LB1B3M	0.001

**BIN C**

379GLBR- 290 Rep 1	18NOV5:309LC1M	10.99
379GLBR- 290 Rep 2	18NOV5:309LC1M	8.30
379GLBR- 290 Rep 3	18NOV5:309LC1M	8.60
379GLBR- 292	18NOV6:1810LC1M	0.31
379GLBR- 293	18NOV6:1810LC1M	2.37
379GLBR- 328	19NOV5:559LC2M	2.16
379GLBR- 333 Rep 1	19NOV5:2010LC2M	4.40
379GLBR- 333 Rep 2	19NOV5:2010LC2M	4.20
379GLBR- 333 Rep 3	19NOV5:2010LC2M	4.19
379GLBR- 334	19NOV5:3510LC2M	1.83
379GLBR- 362	20NOV2:173LC3M	< 0.005
379GLBR- 363	20NOV2:173LC3M	< 0.005
379GLBR- 368	20NOV2:439LC3M	< 0.005
379GLBR- 371	20NOV3:0310LC3M	0.69
379GLBR- 372	20NOV3:0710LC3M	2.92



**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN WATER SAMPLES**

MSL Code	Sponsor ID	Hg (ug/L) CVAA
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**BIN D**

379GLBR- 399	21NOV3:3810LD1M	5.25
379GLBR- 400	21NOV3:4510LD1M	1.48
379GLBR- 405	21NOV4:359LD1M	3.20

**DILUTION WATER**

379GLBR- 277	6NOV12:003LM	0.003
Blank 1		0.017
Blank 2		0.021
Blank 3		0.047

**STANDARD REFERENCE MATERIAL**

ng/l

SRM 1641b	Rep 1	1.46
SRM 1641b	Rep 2	1.45
SRM 1641b	Rep 3	1.82
SRM 1641b	Rep 4	1.59
SRM 1641b	Rep 5	1.41
SRM 1641b	Rep 6	1.37
SRM 1641b	Rep 7	1.44
	<b>certified value</b>	1.52 ±0.04

**MATRIX SPIKE RESULTS**

Amount Spiked		5
379GLBR-290 •	18NOV5:309LC1M	9.30
379GLBR-290 + Spike		15.68
Amount Recovered		6.38
Percent Recovery		128%

Amount Spiked		5
379GLBR-290 •	DUPLICATE	9.30
379GLBR-290 + Spike		17.03
Amount Recovered		7.73
Percent Recovery		155%

**BUFFALO RIVER PILOT PROJECT (CF #379)  
MERCURY IN WATER SAMPLES**

MSL Code	Sponsor ID	Hg (ug/L) CVAA
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**MATRIX SPIKE RESULTS**

Amount Spiked		5
379GLBR-334	19NOV5:3510LC2M	1.83
379GLBR-334+ Spike		7.58
Amount Recovered		5.76
Percent Recovery		115%

Amount Spiked		5
379GLBR-334	DUPLICATE	1.83
379GLBR-334+ Spike		6.39
Amount Recovered		4.56
Percent Recovery		91%

**REPLICATE ANALYSIS**

379GLBR- 290 Rep 1	18NOV5:309LC1M	10.99
379GLBR- 290 Rep 2	18NOV5:309LC1M	8.30
379GLBR- 290 Rep 3	18NOV5:309LC1M	8.60
	RSD %	16%
379GLBR- 333 Rep 1	19NOV5:2010LC2M	4.40
379GLBR- 333 Rep 2	19NOV5:2010LC2M	4.20
379GLBR- 333 Rep 3	19NOV5:2010LC2M	4.19
	RSD %	3%

NOTE: All results are blank-corrected.

\* = Mean of replicated sample.

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Total Solids (% Dry Wt.)	Total Volatile Solids (%Dry Wt.)
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**BIN A**

379GLBR- 39	07OCT12:411SAS	55.19	6.39
379GLBR- 44	07OCT12:321SAS	58.13	6.12
379GLBR- 45	07OCT12:121SAS	60.66	6.63
379GLBR- 54	09OCT11:282SA1S	60.54	5.62
379GLBR- 55	09OCT11:382SA2S	62.97	5.96
379GLBR- 56	09OCT11:502SA3S	60.15	6.03
379GLBR- 98	22OCT11:307SA1S	64.59	6.46
379GLBR- 105	22OCT11:368SA1S	98.45	4.65
379GLBR- 109	22OCT4:203SA2S	52.16	5.84
379GLBR- 110	22OCT5:553SA2S	55.62	5.08
379GLBR- 114	23OCT1:303SA2S	55.69	5.72
379GLBR- 116	23OCT4:254SA2S	99.81	3.44
379GLBR- 117	23OCT4:264SA2S	99.77	3.52
379GLBR- 118	23OCT4:274SA2S	99.78	3.22
379GLBR- 134	24OCT10:457SA2S	95.34	4.69
379GLBR- 137	24OCT10:508SA2S	77.07	2.29
379GLBR- 140 Rep 1	24OCT2:203SA3S	53.65	5.83
379GLBR- 140 Rep 2	24OCT2:203SA3S	53.94	5.96
379GLBR- 140 Rep 3	24OCT2:203SA3S	55.22	6.08
379GLBR- 142	24OCT5:503SA3S	55.79	5.16
379GLBR- 144	24OCT6:203SA3S	56.42	5.02
379GLBR- 147	25OCT9:234SA3S	99.44	4.54
379GLBR- 148	25OCT9:244SA3S	99.45	3.73
379GLBR- 149	25OCT9:254SA3S	99.48	4.46
379GLBR- 157	25OCT9:307SA3S	97.54	5.42
379GLBR- 169	25OCT9:358SA3S	99.57	4.73

**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Total Solids (% Dry Wt.)	Total Volatile Solids (%Dry Wt.)
<b>BIN B</b>			
379GLBR- 36	07OCT1:111SBS	55.89	5.94
379GLBR- 41	07OCT1:021SBS	55.22	5.90
379GLBR- 42	07OCT12:501SBS	54.55	6.62
379GLBR- 62	09OCT9:552SB1S	63.18	5.72
379GLBR- 63	09OCT10:152SB2S	62.04	5.58
379GLBR- 64	09OCT10:352SB3S	63.01	5.61
379GLBR- 172	30OCT2:573SB2S	53.39	4.99
379GLBR- 173	25OCT12:153SB1S	55.20	5.28
379GLBR- 175	25OCT2:153SB1S	56.91	5.80
379GLBR- 178	25OCT2:304SB1S	98.81	3.42
379GLBR- 179	25OCT2:314SB1S	98.67	3.41
379GLBR- 180	25OCT2:324SB1S	98.75	4.22
379GLBR- 191	25OCT3:517SB1S	99.07	5.08
379GLBR- 194	25OCT3:568SB1S	99.51	5.00
379GLBR- 200	30OCT4:053SB2S	55.27	5.73
379GLBR- 205	30OCT4:393SB2S	55.16	5.67
379GLBR- 209	31OCT9:017SB2S	87.07	3.83
379GLBR- 212	31OCT9:068SB2S	99.52	3.79
379GLBR- 216 Rep 1	31OCT9:204SB2S	99.84	2.17
379GLBR- 216 Rep 2	31OCT9:204SB2S	99.84	2.81
379GLBR- 216 Rep 3	31OCT9:204SB2S	99.84	2.91
379GLBR- 217	31OCT9:234SB2S	99.76	2.04
379GLBR- 218	31OCT9:224SB2S	99.71	2.96
379GLBR- 235	31OCT2:364SB3S	99.74	3.90
379GLBR- 236	31OCT11:503SB3S	52.57	5.73
379GLBR- 237	31OCT10:453SB3S	55.34	5.80
379GLBR- 238	31OCT2:303SB3S	52.45	4.68
379GLBR- 239	31OCT2:374SB3S	99.77	4.03
379GLBR- 240	31OCT2:374SB3S	99.75	4.41
379GLBR- 264	1NOV10:208SB3S	99.35	4.39
379GLBR- 266	1NOV10:157SB3S	91.30	4.95

**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Total Solids (% Dry Wt.)	Total Volatile Solids (%Dry Wt.)
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**BIN C.**

379GLBR- 37	07OCT1:2012SCS	54.73	6.30
379GLBR- 38	07OCT1:431SCS	64.58	4.29
379GLBR- 40	07OCT1:331SCS	60.22	4.78
379GLBR- 70	09OCT4:362SC2S	63.85	5.25
379GLBR- 71	09OCT4:552SC1S	63.79	4.91
379GLBR- 72	09OCT4:152SC3S	64.78	5.53
379GLBR- 279	18NOV2:553SC1S	46.95	5.53
379GLBR- 281	18NOV3:503SC1S	54.90	5.29
379GLBR- 283	18NOV4:403SC1S	54.53	5.35
379GLBR- 296	19NOV8:554SC1S	99.84	4.35
379GLBR- 297	19NOV8:564SC1S	99.82	4.38
379GLBR- 298	19NOV8:574SC1S	99.88	4.11
379GLBR- 306	19NOV9:228SC1S	98.37	4.22
379GLBR- 309	19NOV9:337SC1S	82.98	4.88
379GLBR- 310	19NOV11:303SC2S	49.86	4.87
379GLBR- 312	19NOV2:203SC2S	50.34	5.27
379GLBR- 314	19NOV3:553SC2S	52.26	5.56
379GLBR- 317	19NOV4:314SC2S	99.96	4.23
379GLBR- 318	19NOV4:314SC2S	99.96	3.62
379GLBR- 319 Rep 1	19NOV4:314SC2S	100.00	2.90
379GLBR- 319 Rep 2	19NOV4:314SC2S	99.98	4.04
379GLBR- 319 Rep 3	19NOV4:314SC2S	99.99	3.95
379GLBR- 338	20NOV8:157SC2S	95.31	4.87
379GLBR- 339	20NOV11:443SC3S	50.16	5.01
379GLBR- 341	20NOV1:003SC3S	47.26	5.04
379GLBR- 343	20NOV1:553SC3S	47.50	4.83
379GLBR- 347	20NOV3:124SC3S	94.60	3.78
379GLBR- 348	20NOV3:124SC3S	95.77	3.76
379GLBR- 349	20NOV3:124SC3S	95.35	4.77
379GLBR- 355	20NOV3:317SC3S	85.08	5.24
379GLBR- 358	20NOV3:258SC3S	99.57	4.75

**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Total Solids (% Dry Wt.)	Total Volatile Solids (%Dry Wt.)
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**BIN D-**

379GLBR- 34	07OCT2:091SDS	54.45	5.64
379GLBR- 35	07OCT1:521SDS	57.43	5.53
379GLBR- 43	07OCT2:051SDS	58.76	5.21
379GLBR- 48	08OCT5:312SD2S	58.01	5.68
379GLBR- 50	08OCT5:412SD1S	59.33	5.36
379GLBR- 52 Rep 1	08OCT5:172SD3S	57.44	5.98
379GLBR- 52 Rep 2	08OCT5:172SD3S	58.06	6.38
379GLBR- 52 Rep 3	08OCT5:172SD3S	58.14	5.92
379GLBR- 375	21NOV12:003SD1S	40.67	4.09
379GLBR- 377	21NOV1:353SD1S	47.67	5.29
379GLBR- 379	21NOV2:433SD1S	45.34	5.37
379GLBR- 382 Rep 1	21NOV4:558SD1S	99.56	4.48
379GLBR- 382 Rep 2	21NOV4:558SD1S	99.55	4.29
379GLBR- 382 Rep 3	21NOV4:558SD1S	99.57	4.63
379GLBR- 387	21NOV5:087SD1S	94.22	5.11
379GLBR- 392	21NOV5:214SD1S	99.87	3.81
379GLBR- 393	21NOV5:214SD1S	99.89	3.48
379GLBR- 394	21NOV5:214SD1S	99.87	2.79

**BLANK RESULTS**

	Total Solids	Total Volatile Solids
	(concentrations in grams)	
Blank 1	0.0005	0.0009
Blank 2	0.0013	0.0017
Blank 3	0.0001	0.0012
Blank 4	0.0005	0.0008
Blank 5	0.0006	0.0012
Blank 6	0.0005	0.0001
Blank 7	0.0001	0.0005
Blank 8	0.0010	0.0023
Blank 9	0.0014	0.0008
Blank 10	0.0011	0.0025

NOTE: Results are not blank-corrected.

NA = Not applicable/analyzed.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Total Solids (% Dry Wt.)	Total Volatile Solids (%Dry Wt.)
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**REPLICATE ANALYSIS**

379GLBR- 140 Rep 1	24OCT2:203SA3S	53.65	5.83
379GLBR- 140 Rep 2	24OCT2:203SA3S	53.94	5.96
379GLBR- 140 Rep 3	24OCT2:203SA3S	55.22	6.08
	<b>RSD %</b>	<b>2%</b>	<b>2%</b>
379GLBR- 216 Rep 1	31OCT9:204SB2S	99.84	2.17
379GLBR- 216 Rep 2	31OCT9:204SB2S	99.84	2.81
379GLBR- 216 Rep 3	31OCT9:204SB2S	99.84	2.91
	<b>RSD %</b>	<b>0%</b>	<b>15%</b>
379GLBR- 319 Rep 1	19NOV4:314SC2S	100.00	2.9
379GLBR- 319 Rep 2	19NOV4:314SC2S	99.98	4.04
379GLBR- 319 Rep 3	19NOV4:314SC2S	99.99	3.95
	<b>RSD %</b>	<b>0%</b>	<b>17%</b>
379GLBR- 52 Rep 1	08OCT5:172SD3S	57.44	5.98
379GLBR- 52 Rep 2	08OCT5:172SD3S	58.06	6.38
379GLBR- 52 Rep 3	08OCT5:172SD3S	58.14	5.92
	<b>RSD %</b>	<b>1%</b>	<b>4%</b>
379GLBR- 382 Rep 1	21NOV4:558SD1S	99.56	4.48
379GLBR- 382 Rep 2	21NOV4:558SD1S	99.55	4.29
379GLBR- 382 Rep 3	21NOV4:558SD1S	99.57	4.63
	<b>RSD %</b>	<b>0%</b>	<b>4%</b>

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN WATER SAMPLES**

MSL Code	Sponsor ID	Total Solids (mg/L)	Total Suspended Solids (mg/L)
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**BIN A**

379GLBR- 95	21OCT:6.2410LA1S	4214 *	318
379GLBR- 100	21OCT6:459LA1S	7690 *	244
379GLBR- 126	23OCT4:459LA2S	10486 *	344
379GLBR- 130 Rep 1	23OCT5:2010LA2S	7824 *	4947
379GLBR- 130 Rep 2	23OCT5:2010LA2S	8196 *	4810
379GLBR- 130 Rep 3	23OCT5:2010LA2S	8054 *	5113
379GLBR- 158	25OCT10:2210LA3S	12386 *	11080 *
379GLBR- 159	25OCT10:2110LA3S	3564 *	2512 *
379GLBR- 202	30OCT4:166LA3S	184 *	23 *

**BIN B**

379GLBR- 197 Rep 1	25OCT3:1110LB1S	7626 *	7368 *
379GLBR- 197 Rep 2	25OCT3:1110LB1S	7910 *	7040 *
379GLBR- 197 Rep 3	25OCT3:1110LB1S	7674 *	7384 *
379GLBR- 224	31OCT10:059LB2S	6768	216
379GLBR- 230	31OCT10:1910LB2S	2016	616
379GLBR- 258	31OCT2:5410LB3S	990	950
379GLBR- 259	31OCT2:5610LB3S	1156	972
379GLBR- 260	31OCT3:049LB3S	6482	528
379GLBR- 261	31OCT3:059LB3S	6810	776

**BIN C**

379GLBR- 289 Rep 1	18NOV5:409LC1S	11410 *	4748 *
379GLBR- 289 Rep 2	18NOV5:409LC1S	8324 *	5376 *
379GLBR- 289 Rep 3	18NOV5:409LC1S	8164 *	3878 *
379GLBR- 291	18NOV6:1010LC1S	2172 *	1273 *
379GLBR- 329	19NOV5:539LC2S	4918 *	3184 *
379GLBR- 332	19NOV5:2410LC2S	1248 *	863 *
379GLBR- 364	20NOV2:163LC3S	386 *	<1 *
379GLBR- 367	20NOV2:409LC3S	9420 *	8760 *
379GLBR- 373	20NOV3:0010LC3S	996 *	638 *



**BUFFALO RIVER PILOT PROJECT (CF#379)  
SOLIDS IN WATER SAMPLES**

MSL Code	Sponsor ID	Total Solids (mg/L)	Total Suspended Solids (mg/L)
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**BIN D**

379GLBR- 401	21NOV3:3510LD1S	1848	1156
379GLBR- 403 Rep 1	21NOV4:039LD1S	3908	2736
379GLBR- 403 Rep 2	21NOV4:039LD1S	3954	2614
379GLBR- 403 Rep 3	21NOV4:039LD1S	3970	2634

**DILUTION WATER**

379GLBR- 276	6NOV12:003LS	166	8
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**REPLICATE ANALYSES**

379GLBR- 130 Rep 1	23OCT5:2010LA2S	7824 *	5143
379GLBR- 130 Rep 2	23OCT5:2010LA2S	8196 *	5007
379GLBR- 130 Rep 3	23OCT5:2010LA2S	8054 *	5310
	<b>RSD %</b>	<b>2%</b>	<b>3%</b>
379GLBR- 197 Rep 1	25OCT3:1110LB1S	7626 *	7256 *
379GLBR- 197 Rep 2	25OCT3:1110LB1S	7910 *	6928 *
379GLBR- 197 Rep 3	25OCT3:1110LB1S	7674 *	7272 *
	<b>RSD %</b>	<b>2%</b>	<b>3%</b>
379GLBR- 289 Rep 1	18NOV5:409LC1S	11410 *	2580 *
379GLBR- 289 Rep 2	18NOV5:409LC1S	8324 *	2818 *
379GLBR- 289 Rep 3	18NOV5:409LC1S	8164 *	2282 *
	<b>RSD %</b>	<b>20%</b>	<b>10%</b>
379GLBR- 403 Rep 1	21NOV4:039LD1S	3908	2700
379GLBR- 403 Rep 2	21NOV4:039LD1S	3954	2578
379GLBR- 403 Rep 3	21NOV4:039LD1S	3970	2598
	<b>RSD %</b>	<b>1%</b>	<b>2%</b>

NOTE: All results are blank-corrected.

RSD% = Relative Standard Deviation.

\* = Reruns outside holding times.

**BUFFALO RIVER PILOT PROJECT (CF#379)**  
**TOC ANALYSIS OF SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	TOC
MDL		% Dry Weight 300 ug/g

**BIN A**

379GLBR- 39	07OCT12:411SAS	1.91
379GLBR- 44	07OCT12:321SAS	1.77
379GLBR- 45	07OCT12:121SAS	1.91
379GLBR- 54	09OCT11:282SA1S	1.86
379GLBR- 55	09OCT11:382SA2S	1.84
379GLBR- 56	09OCT11:502SA3S	1.90
379GLBR- 98	22OCT11:307SA1S	2.73
379GLBR- 105	22OCT11:368SA1S	1.86
379GLBR- 109	22OCT4:203SA2S	1.87
379GLBR- 110	22OCT5:553SA2S	1.86
379GLBR- 114	23OCT1:303SA2S	1.89
379GLBR- 116	23OCT4:254SA2S	1.25
379GLBR- 117	23OCT4:264SA2S	1.18
379GLBR- 118	23OCT4:274SA2S	1.21
379GLBR- 134	24OCT10:457SA2S	1.48
379GLBR- 137	24OCT10:508SA2S	1.04
379GLBR- 140 Rep 1	24OCT2:203SA3S	1.86
379GLBR- 140 Rep 2	24OCT2:203SA3S	1.99
379GLBR- 140 Rep 3	24OCT2:203SA3S	1.85
379GLBR- 142	24A OCT5:503SA3S	1.90
379GLBR- 144	24OCT6:203SA3S	1.82
379GLBR- 147	25OCT9:234SA3S	1.40
379GLBR- 148	25OCT9:244SA3S	1.47
379GLBR- 149	25OCT9:254SA3S	1.54
379GLBR- 157	25OCT9:307SA3S	1.72
379GLBR- 169	25OCT9:358SA3S	1.47

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	TOC
MDL		% Dry Weight 300 ug/g

**BIN B**

379GLBR- 36	07OCT1:111SBS	1.93
379GLBR- 41	07OCT1:021SBS	1.67
379GLBR- 42	07OCT12:501SBS	1.91
379GLBR- 62	09OCT9:552SB1S	1.82
379GLBR- 63	09OCT10:152SB2S	1.87
379GLBR- 64	09OCT10:352SB3S	1.94
379GLBR- 172	30OCT2:573SB2S	2.14
379GLBR- 173	25OCT12:153SB1S	1.94
379GLBR- 175	25OCT2:153SB1S	1.84
379GLBR- 178	25OCT2:304SB1S	1.49
379GLBR- 179	25OCT2:314SB1S	1.40
379GLBR- 180	25OCT2:324SB1S	1.37
379GLBR- 191	25OCT3:517SB1S	1.71
379GLBR- 194	25OCT3:568SB1S	1.69
379GLBR- 200	30OCT4:053SB2S	1.81
379GLBR- 205	30OCT4:393SB2S	1.79
379GLBR- 209	31OCT9:017SB2S	1.59
379GLBR- 212	31OCT9:068SB2S	1.53
379GLBR- 216 Rep 1	31OCT9:204SB2S	0.99
379GLBR- 216 Rep 2	31OCT9:204SB2S	1.02
379GLBR- 216 Rep 3	31OCT9:204SB2S	1.13
379GLBR- 217	31OCT9:234SB2S	1.02
379GLBR- 218	31OCT9:224SB2S	1.01
379GLBR- 235	31OCT2:364SB3S	1.52
379GLBR- 236	31OCT11:503SB3S	1.80
379GLBR- 237	31OCT10:453SB3S	2.01
379GLBR- 238	31OCT2:303SB3S	1.78
379GLBR- 239	31OCT2:374SB3S	1.39
379GLBR- 240	31OCT2:374SB3S	1.39
379GLBR- 264	1NOV10:208SB3S	1.46
379GLBR- 266	1NOV10:157SB3S	1.42

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	TOC
MDL		% Dry Weight 300 ug/g

**BIN C**

379GLBR- 37	07OCT1:201SCS	1.80
379GLBR- 38	07OCT1:431SCS	1.62
379GLBR- 40	07OCT1:331SCS	1.89
379GLBR- 70	09OCT4:362SC2S	1.77
379GLBR- 71	09OCT4:552SC1S	1.80
379GLBR- 72	09OCT4:152SC3S	1.84
379GLBR- 279	18NOV2:553SC1S	1.84
379GLBR- 281	18NOV3:503SC1S	1.82
379GLBR- 283	18NOV4:403SC1S	1.83
379GLBR- 296	19NOV8:554SC1S	1.49
379GLBR- 297	19NOV8:564SC1S	1.47
379GLBR- 298	19NOV8:574SC1S	1.61
379GLBR- 306	19NOV9:228SC1S	1.46
379GLBR- 309	19NOV9:337SC1S	2.06
379GLBR- 310	19NOV11:303SC2S	1.78
379GLBR- 312	19NOV2:203SC2S	1.85
379GLBR- 314	19NOV3:553SC2S	1.78
379GLBR- 317	19NOV4:314SC2S	1.49
379GLBR- 318	19NOV4:314SC2S	1.57
379GLBR- 319 Rep 1	19NOV4:314SC2S	1.49
379GLBR- 319 Rep 2	19NOV4:314SC2S	1.59
379GLBR- 319 Rep 3	19NOV4:314SC2S	1.56
379GLBR- 338	20NOV8:157SC2S	1.66
379GLBR- 339	20NOV11:443SC3S	1.76
379GLBR- 341	20NOV1:003SC3S	1.76
379GLBR- 343	20NOV1:553SC3S	1.73
379GLBR- 347	20NOV3:124SC3S	1.67
379GLBR- 348	20NOV3:124SC3S	1.61
379GLBR- 349	20NOV3:124SC3S	1.70
379GLBR- 355	20NOV3:317SC3S	2.05
379GLBR- 358	20NOV3:258SC3S	1.59

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	TOC
MDL		% Dry Weight 300 ug/g

**BIN D**

379GLBR-	34	07OCT2:091SDS	1.78
379GLBR-	35	07OCT1:521SDS	1.76
379GLBR-	43	07OCT2:051SDS	1.74
379GLBR-	48	08OCT5:312SD2S	2.55
379GLBR-	50	08OCT5:412SD1S	1.82
379GLBR-	52 Rep 1	08OCT5:172SD3S	1.91
379GLBR-	52 Rep 2	08OCT5:172SD3S	1.98
379GLBR-	52 Rep 3	08OCT5:172SD3S	1.91
379GLBR-	375	21NOV12:003SD1S	1.85
379GLBR-	377	21NOV1:353SD1S	1.84
379GLBR-	379	21NOV2:433SD1S	1.85
379GLBR-	382 Rep 1	21NOV4:558SD1S	1.53
379GLBR-	382 Rep 2	21NOV4:558SD1S	1.53
379GLBR-	382 Rep 3	21NOV4:558SD1S	1.53
379GLBR-	387	21NOV5:087SD1S	1.84
379GLBR-	392	21NOV5:214SD1S	1.35
379GLBR-	393	21NOV5:214SD1S	1.26
379GLBR-	394	21NOV5:214SD1S	1.36
BLANK	Rep 1		0.006
BLANK	Rep 2		0.005
BLANK	Rep 3		0.005
BLANK	Rep 4		0.005
BLANK	Rep 5		0.003
BLANK	Rep 6		0.005
BLANK	Rep 7		0.003
BLANK	Rep 8		0.004
BLANK	Rep 9		0.005
BLANK	Rep 10		0.003

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	TOC
MDL		% Dry Weight 300 ug/g

**STANDARD REFERENCE MATERIAL**

MESS-1	Rep 1	2.42
MESS-1	Rep 2	2.41
MESS-1	Rep 3	2.37
MESS-1	Rep 4	2.39
MESS-1	Rep 5	2.25
	certified value	2.25
		±0.2

**REPLICATE ANALYSES**

379GLBR- 140	Rep 1	24OCT2:203SA3S	1.86
379GLBR- 140	Rep 2	24OCT2:203SA3S	1.99
379GLBR- 140	Rep 3	24OCT2:203SA3S	1.85
		RSD %	4%
379GLBR- 216	Rep 1	31OCT9:204SB2S	0.99
379GLBR- 216	Rep 2	31OCT9:204SB2S	1.02
379GLBR- 216	Rep 3	31OCT9:204SB2S	1.13
		RSD %	7%
379GLBR- 319	Rep 1	19NOV4:314SC2S	1.49
379GLBR- 319	Rep 2	19NOV4:314SC2S	1.59
379GLBR- 319	Rep 3	19NOV4:314SC2S	1.56
		RSD %	3%
379GLBR- 52	Rep 1	08OCT5:172SD3S	1.91
379GLBR- 52	Rep 2	08OCT5:172SD3S	1.98
379GLBR- 52	Rep 3	08OCT5:172SD3S	1.91
		RSD %	2%
379GLBR- 382	Rep 1	21NOV4:558SD1S	1.53
379GLBR- 382	Rep 2	21NOV4:558SD1S	1.53
379GLBR- 382	Rep 3	21NOV4:558SD1S	1.53
		RSD %	0%

NOTE: Results are blank-corrected.  
RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF WATER SAMPLES**

MSL CODE	SPONSOR ID	TOC (ug/L)
MDL	.	1000 ug/L

**BIN A**

379GLBR- 125	23OCT4:459LA2C	2392600
379GLBR- 129	23OCT5:2010LA2C	853490
379GLBR- 162	25OCT10:2110LA3C	509150
379GLBR- 165	25OCT10:009LA3C	716190
379GLBR- 201	30OCT4:206LA3C	530

**BIN B**

379GLBR- 190	25OCT3:1110LB1C	531430
379GLBR- 225	31OCT10:109LB2C	854650
379GLBR- 231	31OCT10:2410LB2C	747090
379GLBR- 262	31OCT2:5810LB3C	437250
379GLBR- 263	31OCT3:099LB3C	909520
379GLBR- 271	1NOV11:006LB2C	350
379GLBR- 273	1NOV2:506LB1B3C	400

**BIN C**

379GLBR- 294	18NOV6:0610LC1C	275150
379GLBR- 295	18NOV5:459LC1C	829720
379GLBR- 330	19NOV5:579LC2C	715150
379GLBR- 335	19NOV5:4310LC2C	251420
379GLBR- 365	20NOV2:183LC3C	7120
379GLBR- 369	20NOV2:449LC3C	428120
379GLBR- 374	20NOV3:0210LC3C	167150

**BIN D**

379GLBR- 402	21NOV3:3710LD1C	373020
379GLBR- 406	21NOV4:459LD1C	695120

**DILUTION WATER**

379GLBR- 278	6NOV12:003LC	1370
BLANK	Rep 1	< 990
BLANK	Rep 2	< 810
BLANK	Rep 3	< 360

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF WATER SAMPLES**

MSL CODE	SPONSOR ID	TOC (ug/L)
MDL		1000 ug/L

**REPLICATE ANALYSES**

379GLBR- 263 Rep 1	31OCT3:099LB3C	902000
379GLBR- 263 Rep 2	31OCT3:099LB3C	935000
379GLBR- 263 Rep 3	31OCT3:099LB3C	905000
	<b>RSD %</b>	<b>2%</b>
379GLBR- 295 Rep 1	18NOV5:459LC1C	829720
379GLBR- 295 Rep 2	18NOV5:459LC1C	850280
379GLBR- 295 Rep 3	18NOV5:459LC1C	856480
	<b>RSD %</b>	<b>2%</b>
379GLBR- 335 Rep 1	19NOV5:4310LC2C	251420
379GLBR- 335 Rep 2	19NOV5:4310LC2C	240650
379GLBR- 335 Rep 3	19NOV5:4310LC2C	238680
	<b>RSD %</b>	<b>3%</b>
379GLBR- 278 Rep 1	6NOV12:003LC	1370
379GLBR- 278 Rep 2	6NOV12:003LC	1390
379GLBR- 278 Rep 3	6NOV12:003LC	1390
	<b>RSD %</b>	<b>1%</b>

**NOTE:** Results are blank-corrected.  
**RSD %** = Relative Standard Deviation.



**REPORT OF CHEMICAL ANALYSES**

**Volume 2: Leach Test**

**PROJECT: Buffalo River Pilot Project**

**Prepared for:**

**USEPA Great Lakes National Program Office  
Attn: Dr. Steve Garbaciak  
230 S. Dearborn  
Chicago, IL 60604**

# REPORT OF: CHEMICAL ANALYSES

PROJECT: BUFFALO RIVER PILOT PROJECT

DATE: April 23, 1992

ISSUED TO: Dr. Steve Garbaciak  
Technical Project Manager  
USEPA Great Lakes National Program Office  
230 S. Dearborn  
Chicago, IL 60604

CF#: 379GLBR

## INTRODUCTION

This report summarizes the results from analyses performed on pilot study samples which were submitted by the U.S. Army Corps of Engineers Great Lakes Division, Buffalo District.

## SAMPLE CUSTODY

Samples were received in good condition from October 7, 1991 through December 5, 1991. Samples were logged in and stored as specified in the narrative. Samples were analyzed within the holding times specified in the QA plan. Any exceptions are noted in the narrative associated with each analysis.

## LEACH TEST AND TCLP EXTRACTIONS

Twenty-one samples were leached following the sequential batch leach test (SBLT) and the toxicity characteristic leaching procedure (TCLP) provided by the Army Corps of Engineers-Buffalo District. All leach test samples were stored at  $4 \pm 2^\circ\text{C}$  prior to leaching. The SBLT produced four extracts for each sample which were subsampled for each parameter, then analyzed separately for metals, specific conductivity, TOC and pH. Metal and TOC aliquots were acidified upon collection to a  $\text{pH} \leq 2$  with nitric acid and phosphoric acid, respectively. Specific conductivity aliquots were stored at  $4 \pm 2^\circ\text{C}$  until analysis. pH was determined immediately and the aliquots were archived at  $4 \pm 2^\circ\text{C}$ . The TCLP produces one extract for each sample which was then analyzed for metals and pH. The metals aliquot was acidified to a  $\text{pH} \leq 2$  upon collection. pH was determined immediately and the aliquot archived at  $4 \pm 2^\circ\text{C}$ . Twelve samples were archived for possible analysis in the future. Samples for TOC were sent to Analytical Resources, Inc. for analysis by EPA method 415.1 and measured on a Dohrman DC-180 Organic Carbon Analyzer. Specific conductance and pH were determined potentiometrically, following Standard Methods 2510 B and EPA method 150.1, respectively. Cadmium, chromium and lead were analyzed by stabilized temperature graphite furnace, following Battelle SOP# MSL-M-32. Mercury was analyzed by cold vapor atomic fluorescence, following Battelle SOP# MSL-M-27.

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
07OCT12:381SAL	Sediment	Leach Test	379GLBR*5
07OCT12:571SBL	Sediment	Leach Test	379GLBR*7
07OCT12:301SAL	Sediment	Leach Test	379GLBR*8
07OCT11:071SBL	Sediment	Leach Test	379GLBR*10
23OCT4:354SA2M	Sediment	Leach Test	379GLBR*121

25OCT9:104SA3M	Sediment	Leach Test	379GLBR*154
25OCT2:514SB1M	Sediment	Leach Test	379GLBR*185
31OCT9:304SB2M	Sediment	Leach Test	379GLBR*214
31OCT2:404SB3M	Sediment	Leach Test	379GLBR*250
4DEC11:455SA2M	Sediment	Leach Test	379GLBR*460
4DEC11:555SA2M	Sediment	Leach Test	379GLBR*461
4DEC12:055SA2M	Sediment	Leach Test	379GLBR*462
4DEC11:155SA3M	Sediment	Leach Test	379GLBR*463
4DEC11:305SA3M	Sediment	Leach Test	379GLBR*464*
4DEC11:375SA3M	Sediment	Leach Test	379GLBR*465*
4DEC12:505SB2M	Sediment	Leach Test	379GLBR*466
4DEC1:005SB2M	Sediment	Leach Test	379GLBR*467*
4DEC1:155SB2M	Sediment	Leach Test	379GLBR*468*
4DEC1:255SB1/B3M	Sediment	Leach Test	379GLBR*469
4DEC1:305SB1/B3M	Sediment	Leach Test	379GLBR*470*
4DEC1:355SB1/B3M	Sediment	Leach Test	379GLBR*471*

\*The second, third and fourth extracts of these leach test were not analyzed, but archived for possible future analysis as directed by the Army Corps of Engineers-Buffalo District.

#### ARCHIVED SAMPLES

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
07OCT1:451SCL	Sediment	Leach Test	379GLBR*1
07OCT1:501SDL	Sediment	Leach Test	379GLBR*2
07OCT2:061SDL	Sediment	Leach Test	379GLBR*3
07OCT2:151SDL	Sediment	Leach Test	379GLBR*4
07OCT1:401SCL	Sediment	Leach Test	379GLBR*6
07OCT1:141SBL	Sediment	Leach Test	379GLBR*9
07OCT1:261SCL	Sediment	Leach Test	379GLBR*11
07OCT12:451SAL	Sediment	Leach Test	379GLBR*12
21OCT5:504SA1L	Sediment	Leach Test	379GLBR*87
19NOV8:504SC1M	Sediment	Leach Test	379GLBR*301
19NOV4:374SC2M	Sediment	Leach Test	379GLBR*322
20NOV3:204SC3M	Sediment	Leach Test	379GLBR*354

#### Metals

The SBLT samples could not be spiked for metals prior to extraction as at a pH >2, metals tend to remain in the sediment. Therefore, any matrix spike added would adsorb onto the sediment and recoveries would be meaningless. Although two TCLP samples (379GLBR\*5 and 465) were spiked with 40 µg of Pb, 20 µg of Cu and 20 µg of Cr prior to extraction, the pH in the extraction fluid was ~ 5, consequently, the metal spikes were not recovered. The analyst performed spike additions at the time of analysis for both the SBLT and TCLP samples. Since spike additions are not true matrix spikes, that data is not presented on the final summary tables but is available in the raw data. Replicate analyses were quite variable for the metals analysis of both TCLP and SBLT extracts, which may be due in part to nonhomogeneous samples and partly to values near the method detection limits. Variability increased for the treated sediment (ash) samples and may be a result of matrix interference. Certified reference material values were always within the ARCS criteria of ± 20%.

Replicates of the leaching procedure for TOC produced variable results, of which some were outside the ARCS criteria. Replicates performed at the time of analysis produced RSD values within the ARCS criteria, indicating the variability comes from the leach procedure itself, rather than the TOC methodology. Generally, the first fraction results were consistent with an RSD ~ 15%, suggesting the variability in extraction efficiency lies in the following days of the method. Variability increased for the treated sediment (ash) samples and may be a result of matrix interference.

MSL Code	Sponsor ID	pH
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379GLBR-	5 Rep 1	Fraction 1	07OCT12:381SAL	7.74
		Fraction 2	07OCT12:381SAL	7.83
		Fraction 3	07OCT12:381SAL	7.93
		Fraction 4	07OCT12:381SAL	7.99
379GLBR-	5 Rep 2	Fraction 1	07OCT12:381SAL	7.75
		Fraction 2	07OCT12:381SAL	7.93
		Fraction 3	07OCT12:381SAL	8.06
		Fraction 4	07OCT12:381SAL	7.97
379GLBR-	5 Rep 3	Fraction 1	07OCT12:381SAL	7.63
		Fraction 2	07OCT12:381SAL	7.93
		Fraction 3	07OCT12:381SAL	8.05
		Fraction 4	07OCT12:381SAL	8.01
379GLBR-	8	Fraction 1	07OCT12:301SAL	6.80
		Fraction 2	07OCT12:301SAL	7.20
		Fraction 3	07OCT12:301SAL	7.56
		Fraction 4	07OCT12:301SAL	7.89
379GLBR- 121	Rep 1	Fraction 1	23OCT4:354SA2M	7.66
		Fraction 2	23OCT4:354SA2M	7.63
		Fraction 3	23OCT4:354SA2M	7.82
		Fraction 4	23OCT4:354SA2M	7.80
379GLBR- 121	Rep 2	Fraction 1	23OCT4:354SA2M	7.57
		Fraction 2	23OCT4:354SA2M	7.87
		Fraction 3	23OCT4:354SA2M	7.68
		Fraction 4	23OCT4:354SA2M	7.80
379GLBR- 121	Rep 3	Fraction 1	23OCT4:354SA2M	7.69
		Fraction 2	23OCT4:354SA2M	7.84
		Fraction 3	23OCT4:354SA2M	7.77
		Fraction 4	23OCT4:354SA2M	7.83
379GLBR- 154		Fraction 1	25OCT9:104SA3M	7.38
		Fraction 2	25OCT9:104SA3M	7.51
		Fraction 3	25OCT9:104SA3M	7.82
		Fraction 4	25OCT9:104SA3M	8.04

**BUFFALO RIVER PILOT PROJECT (CF#379)  
pH ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code		Sponsor ID	pH
379GLBR- 460	Fraction 1	4DEC11:455SA2M	12.05
	Fraction 2	4DEC11:455SA2M	12.38
	Fraction 3	4DEC11:455SA2M	12.27
	Fraction 4	4DEC11:455SA2M	11.80
379GLBR- 461	Fraction 1	4DEC11:555SA2M	12.01
	Fraction 2	4DEC11:555SA2M	12.42
	Fraction 3	4DEC11:555SA2M	12.32
	Fraction 4	4DEC11:555SA2M	11.95
379GLBR- 462	Fraction 1	4DEC12:055SA2M	12.08
	Fraction 2	4DEC12:055SA2M	12.40
	Fraction 3	4DEC12:055SA2M	12.31
	Fraction 4	4DEC12:055SA2M	11.98
379GLBR- 463	Fraction 1	4DEC11:155SA3M	12.45
	Fraction 2	4DEC11:155SA3M	12.50
	Fraction 3	4DEC11:155SA3M	11.02
	Fraction 4	4DEC11:155SA3M	11.03
379GLBR- 464	Fraction 1	4DEC11:305SA3M	12.28
379GLBR- 465	Fraction 1	4DEC11:375SA3M	12.26
<b>BIN B</b>			
379GLBR- 7	Fraction 1	07OCT12:571SBL	7.47
	Fraction 2	07OCT12:571SBL	7.79
	Fraction 3	07OCT12:571SBL	7.80
	Fraction 4	07OCT12:571SBL	7.68
379GLBR- 10	Fraction 1	07OCT1:071SBL	7.92
	Fraction 2	07OCT1:071SBL	7.84
	Fraction 3	07OCT1:071SBL	7.78
	Fraction 4	07OCT1:071SBL	7.79
379GLBR- 214	Fraction 1	31OCT9:304SB2M	7.34
	Fraction 2	31OCT9:304SB2M	7.73
	Fraction 3	31OCT9:304SB2M	7.96
	Fraction 4	31OCT9:304SB2M	8.06
379GLBR- 250	Fraction 1	31OCT2:404SB3M	7.32
	Fraction 2	31OCT2:404SB3M	7.53
	Fraction 3	31OCT2:404SB3M	7.72
	Fraction 4	31OCT2:404SB3M	7.82

**BUFFALO RIVER PILOT PROJECT (CF#379)**  
**pH ANALYSIS OF LEACHATE SAMPLES**  
**(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	pH
379GLBR- 466	Fraction 1 4DEC12:505SB2M	12.19
	Fraction 2 4DEC12:505SB2M	12.48
	Fraction 3 4DEC12:505SB2M	12.44
	Fraction 4 4DEC12:505SB2M	12.27
379GLBR- 467	Fraction 1 4DEC1:005SB2M	12.32
379GLBR- 468	Fraction 1 4DEC1:155SB2M	12.30
379GLBR- 469	Fraction 1 4DEC1:255SB1/B3M	11.93
	Fraction 2 4DEC1:255SB1/B3M	12.20
	Fraction 3 4DEC1:255SB1/B3M	12.08
	Fraction 4 4DEC1:255SB1/B3M	11.98
379GLBR- 470	Fraction 1 4DEC1:305SB1/B3M	12.42
379GLBR- 471	Fraction 1 4DEC1:355SB1/B3M	12.43
BLANK-1	Fraction 1	5.60
BLANK-1	Fraction 2	5.75
BLANK-1	Fraction 3	5.63
BLANK-1	Fraction 4	6.00
BLANK-2	Fraction 1	6.85
BLANK-2	Fraction 2	6.25
BLANK-2	Fraction 3	5.04
BLANK-2	Fraction 4	5.18
BLANK-3	Fraction 1	5.32
BLANK-3	Fraction 2	4.89
BLANK-3	Fraction 3	5.07
BLANK-3	Fraction 4	5.57

**BUFFALO RIVER PILOT PROJECT (CF#379)  
pH ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	pH
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**REPLICATE ANALYSIS**

379GLBR-	5 Rep 1 Fraction 1	07OCT12:381SAL	7.74
379GLBR-	5 Rep 2 Fraction 1	07OCT12:381SAL	7.75
379GLBR-	5 Rep 3 Fraction 1	07OCT12:381SAL	7.63
		RSD %	1%
379GLBR-	5 Rep 1 Fraction 2	07OCT12:381SAL	7.83
379GLBR-	5 Rep 2 Fraction 2	07OCT12:381SAL	7.93
379GLBR-	5 Rep 3 Fraction 2	07OCT12:381SAL	7.93
		RSD %	1%
379GLBR-	5 Rep 1 Fraction 3	07OCT12:381SAL	7.93
379GLBR-	5 Rep 2 Fraction 3	07OCT12:381SAL	8.06
379GLBR-	5 Rep 3 Fraction 3	07OCT12:381SAL	8.05
		RSD %	1%
379GLBR-	5 Rep 1 Fraction 4	07OCT12:381SAL	7.99
379GLBR-	5 Rep 2 Fraction 4	07OCT12:381SAL	7.97
379GLBR-	5 Rep 3 Fraction 4	07OCT12:381SAL	8.01
		RSD %	0%
379GLBR- 121	Rep 1 Fraction 1	23OCT4:354SA2M	7.66
379GLBR- 121	Rep 2 Fraction 1	23OCT4:354SA2M	7.57
379GLBR- 121	Rep 3 Fraction 1	23OCT4:354SA2M	7.69
		RSD %	1%
379GLBR- 121	Rep 1 Fraction 2	23OCT4:354SA2M	7.63
379GLBR- 121	Rep 2 Fraction 2	23OCT4:354SA2M	7.87
379GLBR- 121	Rep 3 Fraction 2	23OCT4:354SA2M	7.84
		RSD %	2%

RSD % = Relative Standard Deviation.



**BUFFALO RIVER PILOT PROJECT (CF#379)  
pH ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	pH
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**REPLICATE ANALYSIS**

379GLBR- 121 Rep 1 Fraction 3	23OCT4:354SA2M	7.82
379GLBR- 121 Rep 2 Fraction 3	23OCT4:354SA2M	7.68
379GLBR- 121 Rep 3 Fraction 3	23OCT4:354SA2M	7.77
	<b>RSD %</b>	<b>1%</b>
379GLBR- 121 Rep 1 Fraction 4	23OCT4:354SA2M	7.80
379GLBR- 121 Rep 2 Fraction 4	23OCT4:354SA2M	7.80
379GLBR- 121 Rep 3 Fraction 4	23OCT4:354SA2M	7.83
	<b>RSD %</b>	<b>0%</b>

**RSD % = Relative Standard Deviation.**

**BUFFALO RIVER PILOT PROJECT (CF#379)  
pH ANALYSIS OF LEACHATE SAMPLES  
(TCLP EXTRACTONS)**

MSL Code	Sponsor ID	pH
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**BIN A**

379GLBR- 5 Rep 1	07OCT12:381SAL	5.53
379GLBR- 5 Rep 2	07OCT12:381SAL	5.54
379GLBR- 5 Rep 3	07OCT12:381SAL	5.56
379GLBR- 8	07OCT12:301SAL	5.55
379GLBR- 121 Rep 1	23OCT4:354SA2M	6.24
379GLBR- 121 Rep 2	23OCT4:354SA2M	6.22
379GLBR- 121 Rep 3	23OCT4:354SA2M	6.17
379GLBR- 154	25OCT9:104SA3M	6.10
379GLBR- 460	4DEC11:455SA2M	11.71
379GLBR- 461	4DEC11:555SA2M	11.84
379GLBR- 462	4DEC12:055SA2M	9.28
379GLBR- 463	4DEC11:155SA3M	11.64
379GLBR- 464	4DEC11:305SA3M	11.71
379GLBR- 465	4DEC11:375SA3M	11.67

**BIN B**

379GLBR- 7	07OCT12:571SBL	5.53
379GLBR- 10	07OCT1:071SBL	5.58
379GLBR- 214	31OCT9:304SB2M	6.23
379GLBR- 250	31OCT2:404SB3M	6.10
379GLBR- 466	4DEC12:505SB2M	12.07
379GLBR- 467	4DEC1:005SB2M	12.05
379GLBR- 468	4DEC1:155SB2M	12.05
379GLBR- 469	4DEC1:255SB1/B3M	6.64
379GLBR- 470	4DEC1:305SB1/B3M	6.55
379GLBR- 471	4DEC1:355SB1/B3M	6.64
BLANK-1		4.92
BLANK-2		2.86

**BUFFALO RIVER PILOT PROJECT (CF#379)  
pH ANALYSIS OF LEACHATE SAMPLES  
(TCLP EXTRACTONS)**

MSL Code	Sponsor ID	pH
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**REPLICATE ANALYSIS**

379GLBR- 5 Rep 1	07OCT12:381SAL	5.53
379GLBR- 5 Rep 2	07OCT12:381SAL	5.54
379GLBR- 5 Rep 3	07OCT12:381SAL	5.56
	<b>RSD %</b>	<b>0%</b>
379GLBR- 121 Rep 1	23OCT4:354SA2M	6.24
379GLBR- 121 Rep 2	23OCT4:354SA2M	6.22
379GLBR- 121 Rep 3	23OCT4:354SA2M	6.17
	<b>RSD %</b>	<b>1%</b>

**RSD % = Relative Standard Deviation.**

**BUFFALO RIVER PILOT PROJECT (CF #379)  
SPECIFIC CONDUCTANCE IN LEACHATE SAMPLES**

MSL Code	Fraction No.	Sponsor ID	Specific Conductance (umhos/cm)
BIN A			
379GLBR-5, Rep 1	Fraction 1	07OCT12:381SAL	440
	Fraction 2	07OCT12:381SAL	230
	Fraction 3	07OCT12:381SAL	162
	Fraction 4	07OCT12:381SAL	136
379GLBR-5, Rep 2	Fraction 1	07OCT12:381SAL	450
	Fraction 2	07OCT12:381SAL	220
	Fraction 3	07OCT12:381SAL	164
	Fraction 4	07OCT12:381SAL	136
379GLBR-5, Rep 3	Fraction 1	07OCT12:381SAL	480
	Fraction 2	07OCT12:381SAL	230
	Fraction 3	07OCT12:381SAL	171
	Fraction 4	07OCT12:381SAL	134
379GLBR-8	Fraction 1	07OCT12:301SAL	740
	Fraction 2	07OCT12:301SAL	570
	Fraction 3	07OCT12:301SAL	340
	Fraction 4	07OCT12:301SAL	240
379GLBR-121, Rep 1	Fraction 1	23OCT4:354SA2M	1030
	Fraction 2	23OCT4:354SA2M	230
	Fraction 3	23OCT4:354SA2M	103
	Fraction 4	23OCT4:354SA2M	89
379GLBR-121, Rep 2	Fraction 1	23OCT4:354SA2M	1010
	Fraction 2	23OCT4:354SA2M	230
	Fraction 3	23OCT4:354SA2M	109
	Fraction 4	23OCT4:354SA2M	88
379GLBR-121, Rep 3	Fraction 1	23OCT4:354SA2M	980
	Fraction 2	23OCT4:354SA2M	220
	Fraction 3	23OCT4:354SA2M	104
	Fraction 4	23OCT4:354SA2M	87
379GLBR-154	Fraction 1	25OCT9:104SA3M	750
	Fraction 2	25OCT9:104SA3M	310
	Fraction 3	25OCT9:104SA3M	210
	Fraction 4	25OCT9:104SA3M	154
379GLBR-460	Fraction 1	4DEC11:455SA2M	6960
	Fraction 2	4DEC11:455SA2M	7080
	Fraction 3	4DEC11:455SA2M	5400
	Fraction 4	4DEC11:455SA2M	4160

**BUFFALO RIVER PILOT PROJECT (CF #379)  
SPECIFIC CONDUCTANCE IN LEACHATE SAMPLES**

MSL Code	Fraction No.	Sponsor ID	Specific Conductance (umhos/cm)
<b>BIN A</b>			
379GLBR-461	Fraction 1	4DEC11:555SA2M	7560
	Fraction 2	4DEC11:555SA2M	6960
	Fraction 3	4DEC11:555SA2M	6240
	Fraction 4	4DEC11:555SA2M	4560
379GLBR-462	Fraction 1	4DEC12:055SA2M	7480
	Fraction 2	4DEC12:055SA2M	6840
	Fraction 3	4DEC12:055SA2M	5640
	Fraction 4	4DEC12:055SA2M	3960
379GLBR-463	Fraction 1	4DEC11:155SA3M	9040
	Fraction 2	4DEC11:155SA3M	8720
	Fraction 3	4DEC11:155SA3M	8880
	Fraction 4	4DEC11:155SA3M	8160
379GLBR-464	Fraction 1	4DEC11:305SA3M	9600
379GLBR-465	Fraction 1	4DEC11:375SA3M	9680
<b>BIN B</b>			
379GLBR-7	Fraction 1	07OCT1:571SBL	480
	Fraction 2	07OCT1:571SBL	230
	Fraction 3	07OCT1:571SBL	174
	Fraction 4	07OCT1:571SBL	200
379GLBR-214	Fraction 1	31OCT9:304SB2M	1100
	Fraction 2	31OCT9:304SB2M	250
	Fraction 3	31OCT9:304SB2M	116
	Fraction 4	31OCT9:304SB2M	88
379GLBR-466	Fraction 1	4DEC12:505SB2M	10160
	Fraction 2	4DEC12:505SB2M	8720
	Fraction 3	4DEC12:505SB2M	8080
	Fraction 4	4DEC12:505SB2M	8000
379GLBR-467	Fraction 1	4DEC1:005SB2M	10240
379GLBR-468	Fraction 1	4DEC1:155SB2M	11280
379GLBR-469	Fraction 1	4DEC1:2555SB1/B3M	6600
	Fraction 2	4DEC1:2555SB1/B3M	5400
	Fraction 3	4DEC1:2555SB1/B3M	3320
	Fraction 4	4DEC1:2555SB1/B3M	2740
379GLBR-470	Fraction 1	4DEC1:305SB1/B3M	6720
379GLBR-471	Fraction 1	4DEC1:355SB1/B3M	7400

**BUFFALO RIVER PILOT PROJECT (CF #379)  
SPECIFIC CONDUCTANCE IN LEACHATE SAMPLES**

MSL Code	Fraction No.	Sponsor ID	Specific Conductance (umhos/cm)
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BLANK 1	Fraction 1		3
BLANK 1	Fraction 2		4
BLANK 1	Fraction 3		2
BLANK 1	Fraction 4		2
BLANK 2	Fraction 1		3
BLANK 2	Fraction 2		3
BLANK 2	Fraction 3		8
BLANK 2	Fraction 4		5
BLANK 3	Fraction 1		13
BLANK 3	Fraction 2		7
BLANK 3	Fraction 3		5
BLANK 3	Fraction 4		7

**STANDARD - KC2 (umhos/cm)**

.01M	Rep 1	1410
.01M	Rep 2	1410
.01M	Rep 3	1410
.01M	Rep 4	1410
	TRUE VALUE	1413

**STANDARD - KC1 (umhos/cm)**

.005M	Rep 1	730
.005M	Rep 2	730
.005M	Rep 3	730
.005M	Rep 4	730
	TRUE VALUE	717.8

**REPLICATE ANALYSIS**

379GLBR-5, Rep 1	Fraction 1	07OCT12:381SAL	440
379GLBR-5, Rep 2	Fraction 1	07OCT12:381SAL	450
379GLBR-5, Rep 3	Fraction 1	07OCT12:381SAL	480
	RSD %		5%

**BUFFALO RIVER PILOT PROJECT (CF #379)  
SPECIFIC CONDUCTANCE IN LEACHATE SAMPLES**

MSL Code	Fraction No.	Sponsor ID	Specific Conductance (umhos/cm)
<b>REPLICATE ANALYSIS</b>			
379GLBR-5, Rep 1	Fraction 2	07OCT12:381SAL	230
379GLBR-5, Rep 2	Fraction 2	07OCT12:381SAL	220
379GLBR-5, Rep 3	Fraction 2	07OCT12:381SAL	230
		<b>RSD %</b>	<b>3%</b>
379GLBR-5, Rep 1	Fraction 3	07OCT12:381SAL	162
379GLBR-5, Rep 2	Fraction 3	07OCT12:381SAL	164
379GLBR-5, Rep 3	Fraction 3	07OCT12:301SAL	171
		<b>RSD %</b>	<b>3%</b>
379GLBR-5, Rep 1	Fraction 4	07OCT12:381SAL	136
379GLBR-5, Rep 2	Fraction 4	07OCT12:381SAL	136
379GLBR-5, Rep 3	Fraction 4	07OCT12:301SAL	134
		<b>RSD %</b>	<b>1%</b>
379GLBR-121, Rep 1	Fraction 1	23OCT4:354SA2M	1030
379GLBR-121, Rep 2	Fraction 1	23OCT4:354SA2M	1010
379GLBR-121, Rep 3	Fraction 1	23OCT4:354SA2M	980
		<b>RSD %</b>	<b>2%</b>
379GLBR-121, Rep 1	Fraction 2	23OCT4:354SA2M	230
379GLBR-121, Rep 2	Fraction 2	23OCT4:354SA2M	230
379GLBR-121, Rep 3	Fraction 2	23OCT4:354SA2M	220
		<b>RSD %</b>	<b>3%</b>
379GLBR-121, Rep 1	Fraction 3	23OCT4:354SA2M	103
379GLBR-121, Rep 2	Fraction 3	23OCT4:354SA2M	109
379GLBR-121, Rep 3	Fraction 3	23OCT4:354SA2M	104
		<b>RSD %</b>	<b>3%</b>
379GLBR-121, Rep 1	Fraction 4	23OCT4:354SA2M	89
379GLBR-121, Rep 2	Fraction 4	23OCT4:354SA2M	88
379GLBR-121, Rep 3	Fraction 4	23OCT4:354SA2M	87
		<b>RSD %</b>	<b>1%</b>

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF#379)**  
**TOC ANALYSIS OF LEACHATE SAMPLES**  
**(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	TOC ug/L
Method Detection Limit		660

**BIN A**

379GLBR-	5 Rep 1	Fraction 1	07OCT12:381SAL	10,620
		Fraction 2	07OCT12:381SAL	10,230
		Fraction 3	07OCT12:381SAL	7,160
		Fraction 4	07OCT12:381SAL	5,710
379GLBR-	5 Rep 2	Fraction 1	07OCT12:381SAL	10,880
		Fraction 2	07OCT12:381SAL	7,760
		Fraction 3	07OCT12:381SAL	6,310
		Fraction 4	07OCT12:381SAL	4,170
379GLBR-	5 Rep 3	Fraction 1	07OCT12:381SAL	13,370
		Fraction 2	07OCT12:381SAL	8,940
		Fraction 3	07OCT12:381SAL	10,300
		Fraction 4	07OCT12:381SAL	5,010
379GLBR-	8	Fraction 1	07OCT12:301SAL	17,350
		Fraction 2	07OCT12:301SAL	11,720
		Fraction 3	07OCT12:301SAL	12,070
		Fraction 4	07OCT12:301SAL	4,680
379GLBR-	121 Rep 1	Fraction 1	23OCT4:354SA2M	34,150
		Fraction 2	23OCT4:354SA2M	15,660
		Fraction 3	23OCT4:354SA2M	3,560
		Fraction 4	23OCT4:354SA2M	12,510
379GLBR-	121 Rep 2	Fraction 1	23OCT4:354SA2M	35,460
		Fraction 2	23OCT4:354SA2M	7,160
		Fraction 3	23OCT4:354SA2M	4,410
		Fraction 4	23OCT4:354SA2M	3,690
379GLBR-	121 Rep 3	Fraction 1	23OCT4:354SA2M	36,550
		Fraction 2	23OCT4:354SA2M	14,150
		Fraction 3	23OCT4:354SA2M	7,040
		Fraction 4	23OCT4:354SA2M	4,440
379GLBR-	154	Fraction 1	25OCT9:104SA3M	136,290
		Fraction 2	25OCT9:104SA3M	32,760
		Fraction 3	25OCT9:104SA3M	20,400
		Fraction 4	25OCT9:104SA3M	12,860



**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	TOC ug/L
Method Detection Limit		660

**BIN A**

379GLBR- 460	Fraction 1	4DEC11:455SA2M	65,200
	Fraction 2	4DEC11:455SA2M	34,300
	Fraction 3	4DEC11:455SA2M	24,600
	Fraction 4	4DEC11:455SA2M	26,600
379GLBR- 461	Fraction 1	4DEC11:555SA2M	70,170
	Fraction 2	4DEC11:555SA2M	33,730
	Fraction 3	4DEC11:555SA2M	23,660
	Fraction 4	4DEC11:555SA2M	18,260
379GLBR- 462	Fraction 1	4DEC12:055SA2M	105,720
	Fraction 2	4DEC12:055SA2M	156,100
	Fraction 3	4DEC12:055SA2M	157,190
	Fraction 4	4DEC12:055SA2M	100,540
379GLBR- 463	Fraction 1	4DEC11:155SA3M	71,100
	Fraction 2	4DEC11:155SA3M	64,900
	Fraction 3	4DEC11:155SA3M	75,090
	Fraction 4	4DEC11:155SA3M	62,430
379GLBR- 464	Fraction 1	4DEC11:305SA3M	56,320
379GLBR- 465	Fraction 1	4DEC11:375SA3M	51,960

**BIN B**

379GLBR- 7	Fraction 1	07OCT12:571SBL	15,830
	Fraction 2	07OCT12:571SBL	4,910
	Fraction 3	07OCT12:571SBL	1,720
	Fraction 4	07OCT12:571SBL	1,740
379GLBR- 10	Fraction 1	07OCT1:071SBL	11,680
	Fraction 2	07OCT1:071SBL	7,120
	Fraction 3	07OCT1:071SBL	5,900
	Fraction 4	07OCT1:071SBL	5,450
379GLBR- 214	Fraction 1	31OCT9:304SB2M	15,400
	Fraction 2	31OCT9:304SB2M	13,800
	Fraction 3	31OCT9:304SB2M	3,960
	Fraction 4	31OCT9:304SB2M	3,360

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	TOC ug/L
Method Detection Limit		660

**BIN B**

379GLBR- 250	Fraction 1	31OCT2:404SB3M	158,670
	Fraction 2	31OCT2:404SB3M	52,600
	Fraction 3	31OCT2:404SB3M	21,510
	Fraction 4	31OCT2:404SB3M	13,410
379GLBR- 466	Fraction 1	4DEC12:505SB2M	108,010
	Fraction 2	4DEC12:505SB2M	134,310
	Fraction 3	4DEC12:505SB2M	121,990
	Fraction 4	4DEC12:505SB2M	97,850
379GLBR- 467	Fraction 1	4DEC1:005SB2M	67,120
379GLBR- 468	Fraction 1	4DEC1:155SB2M	52,820
379GLBR- 469	Fraction 1	4DEC1:255SB1/B3M	88,910
	Fraction 2	4DEC1:255SB1/B3M	38,470
	Fraction 3	4DEC1:255SB1/B3M	26,910
	Fraction 4	4DEC1:255SB1/B3M	23,370
379GLBR- 470	Fraction 1	4DEC1:305SB1/B3M	118,120
379GLBR- 471	Fraction 1	4DEC1:355SB1/B3M	91,590
BLANK-1	Fraction 1		6,680
BLANK-1	Fraction 2		950
BLANK-1	Fraction 3		1,250
BLANK-1	Fraction 4		3,890
BLANK-2	Fraction 1		5,980
BLANK-2	Fraction 2		5,010
BLANK-2	Fraction 3		2,010
BLANK-2	Fraction 4		840
BLANK-3	Fraction 1		9,460
BLANK-3	Fraction 2		1,750
BLANK-3	Fraction 3		1,310
BLANK-3	Fraction 4		1,720

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	TOC ug/L
Method Detection Limit		660

**REPLICATE ANALYSIS**

379GLBR-	5 Rep 1 Fraction 1	07OCT12:381SAL	10,620
379GLBR-	5 Rep 2 Fraction 1	07OCT12:381SAL	10,880
379GLBR-	5 Rep 3 Fraction 1	07OCT12:381SAL	13,370
		RSD %	13%
379GLBR-	5 Rep 1 Fraction 2	07OCT12:381SAL	10,230
379GLBR-	5 Rep 2 Fraction 2	07OCT12:381SAL	7,760
379GLBR-	5 Rep 3 Fraction 2	07OCT12:381SAL	8,940
		RSD %	14%
379GLBR-	5 Rep 1 Fraction 3	07OCT12:381SAL	7,160
379GLBR-	5 Rep 2 Fraction 3	07OCT12:381SAL	6,310
379GLBR-	5 Rep 3 Fraction 3	07OCT12:381SAL	10,300
		RSD %	27%
379GLBR-	5 Rep 1 Fraction 4	07OCT12:381SAL	5,710
379GLBR-	5 Rep 2 Fraction 4	07OCT12:381SAL	4,170
379GLBR-	5 Rep 3 Fraction 4	07OCT12:381SAL	5,010
		RSD %	16%
379GLBR- 121	Rep 1 Fraction 1	23OCT4:354SA2M	34,150
379GLBR- 121	Rep 2 Fraction 1	23OCT4:354SA2M	35,460
379GLBR- 121	Rep 3 Fraction 1	23OCT4:354SA2M	36,550
		RSD %	3%
379GLBR- 121	Rep 1 Fraction 2	23OCT4:354SA2M	15,660
379GLBR- 121	Rep 2 Fraction 2	23OCT4:354SA2M	7,160
379GLBR- 121	Rep 3 Fraction 2	23OCT4:354SA2M	14,150
		RSD %	37%

**BUFFALO RIVER PILOT PROJECT (CF#379)  
TOC ANALYSIS OF LEACHATE SAMPLES  
(SBLT EXTRACTONS)**

MSL Code	Sponsor ID	TOC ug/L
Method Detection Limit		660

**REPLICATE ANALYSIS**

379GLBR- 121 Rep 1 Fraction 3	23OCT4:354SA2M	3,560
379GLBR- 121 Rep 2 Fraction 3	23OCT4:354SA2M	4,410
379GLBR- 121 Rep 3 Fraction 3	23OCT4:354SA2M	7,040
	RSD %	36%
379GLBR- 121 Rep 1 Fraction 4	23OCT4:354SA2M	12,510
379GLBR- 121 Rep 2 Fraction 4	23OCT4:354SA2M	3,690
379GLBR- 121 Rep 3 Fraction 4	23OCT4:354SA2M	4,440
	RSD %	71%

RSD % = Relative Standard Deviation.

NOTE: All results are blank-corrected.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN LEACHATE SAMPLES  
(TCLP SEDIMENT EXTRACTIONS)**

MSL Code	Sponsor ID	Cr (ug/L) AA	Cu (ug/L) AA	Hg (ug/L) CVAF	Pb (ug/L) AA
Method	Detection Limits	0.05	0.85	—	—

**BIN A**

379GLBR- 5 Rep 1	07OCT12:381SAL	0.40	13.6	0.00216	21.95
379GLBR- 5 Rep 2	07OCT12:381SAL	0.36	11.2	0.00094 U	19.88
379GLBR- 5 Rep 3	07OCT12:381SAL	0.36	11.7	0.00094 U	17.39
379GLBR- 8	07OCT12:301SAL	0.62	11.2	0.00094 U	16.15
379GLBR- 121 Rep 1	23OCT4:354SA2M	0.22	5.3	0.00047 U	3.08
379GLBR- 121 Rep 2	23OCT4:354SA2M	0.22	4.4	0.00047 U	3.08
379GLBR- 121 Rep 3	23OCT4:354SA2M	0.36	2.9	0.00094 U	1.54
379GLBR- 154	25OCT9:104SA3M	0.71	15.1	0.00094 U	6.55
379GLBR- 460	4DEC11:455SA2M	42.37	47.6	0.00094 U	0.72 U
379GLBR- 461	4DEC11:555SA2M	36.40	40.8	0.00094 U	0.72 U
379GLBR- 462	4DEC12:055SA2M	30.77	38.4	0.00094 U	0.69 U
379GLBR- 463	4DEC11:155SA3M	50.39	12.6	0.00094 U	0.69 U
379GLBR- 464	4DEC11:305SA3M	45.62	11.2	0.00094 U	0.69 U
379GLBR- 465	4DEC11:375SA3M	37.13	12.1	0.00094 U	0.69 U

**BIN B**

379GLBR- 7	07OCT12:571SBL	0.76	11.2	0.00094 U	16.57
379GLBR- 10	07OCT1:071SBL	0.62	12.1	0.00047 U	19.47
379GLBR- 214	31OCT9:304SB2M	0.13	2.4	0.00094 U	1.93
379GLBR- 250	31OCT2:404SB3M	0.71	13.6	0.00094 U	6.16
379GLBR- 466	4DEC12:505SB2M	18.04	11.7	0.00094 U	0.69 U
379GLBR- 467	4DEC1:005SB2M	17.50	13.1	0.00094 U	0.80
379GLBR- 468	4DEC1:155SB2M	18.04	14.6	0.00094 U	0.69 U
379GLBR- 469	4DEC1:255SB1/B3M	1.91	31.6	0.00996	1.20
379GLBR- 470	4DEC1:305SB1/B3M	2.29	29.6	0.00589	1.20
379GLBR- 471	4DEC1:355SB1/B3M	1.82	31.1	0.00695	0.80
BLANK-1		0.09	1.9	0.00094 U	0.72 U
BLANK-2		0.09	1.0	0.00094 U	0.72 U

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN LEACHATE SAMPLES  
(TCLP SEDIMENT EXTRACTIONS)**

MSL Code	Sponsor ID	Cr (ug/L) AA	Cu (ug/L) AA	Hg (ug/L) CVAF	Pb (ug/L) AA
Method Detection Limits		0.05	0.85	—	—

**STANDARD REFERENCE MATERIAL**

1643c-1	Rep 1	18.90	27.9	NA	36.43
1643c-1	Rep 2	18.37	30.1	NA	35.42
	<b>certified value</b>	19.00	22.3	NA	35.3
		±0.6	±2.8	NA	±0.9
1641b	Rep 1	NA	NA	1372	NA
1641b	Rep 2	NA	NA	1452	NA
1641b	Rep 3	NA	NA	1484	NA
1641b	Rep 4	NA	NA	1596	NA
1641b	Rep 5	NA	NA	1572	NA
1641b	Rep 6	NA	NA	1454	NA
	<b>certified value</b>	NA	NA	1520	NA
		NA	NA	±40	NA

**REPLICATE ANALYSIS**

379GLBR-	5 Rep 1	07OCT12:381SAL	0.40	13.6	0.00216	21.95
379GLBR-	5 Rep 2	07OCT12:381SAL	0.36	11.2	0.00094 U	19.88
379GLBR-	5 Rep 3	07OCT12:381SAL	0.36	11.7	0.00094 U	17.39
		<b>RSD %</b>	<b>6%</b>	<b>10%</b>	<b>NA</b>	<b>12%</b>
379GLBR-	121 Rep 1	23OCT4:354SA2M	0.22	5.3	0.00047 U	3.08
379GLBR-	121 Rep 2	23OCT4:354SA2M	0.22	4.4	0.00047 U	3.08
379GLBR-	121 Rep 3	23OCT4:354SA2M	0.36	2.9	0.00094 U	1.54
		<b>RSD %</b>	<b>30%</b>	<b>29%</b>	<b>NA</b>	<b>35%</b>

U = Detected at or below detection limit.

NA = Not applicable/analyzed.

RSD % = Relative Standard Deviation

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN LEACHATE SAMPLES  
(SBLT SEDIMENT EXTRACTIONS)**

MSL Code	Sponsor ID		Cr (ug/L) AA	Cu (ug/L) AA	Hg (ug/L) CVAF	Pb (ug/L) AA	
BIN A							
379GLBR- 5	Rep 1	Fraction 1	07OCT12:381SAL	0.50	1.9 U	0.00094 U	0.72 U
		Fraction 2	07OCT12:381SAL	0.50	1.9 U	0.00047 U	0.72 U
		Fraction 3	07OCT12:381SAL	0.50	5.2	0.00060	6.63
		Fraction 4	07OCT12:381SAL	0.75	2.8	0.00047 U	4.56
379GLBR- 5	Rep 2	Fraction 1	07OCT12:381SAL	0.50	1.9 U	0.00094 U	1.66
		Fraction 2	07OCT12:381SAL	0.38	1.9 U	0.00047 U	0.83
		Fraction 3	07OCT12:381SAL	0.38	3.6	0.00069	5.80
		Fraction 4	07OCT12:381SAL	1.26	3.2	0.00047 U	3.31
379GLBR- 5	Rep 3	Fraction 1	07OCT12:381SAL	0.50	1.9 U	0.00047 U	1.24
		Fraction 2	07OCT12:381SAL	0.76	1.9 U	0.00047 U	1.66
		Fraction 3	07OCT12:381SAL	0.76	2.4	0.00047 U	4.56
		Fraction 4	07OCT12:381SAL	0.50	2.0	0.00047 U	2.07
379GLBR- 8		Fraction 1	07OCT12:301SAL	0.23 U	5.6	0.00047 U	1.18 U
		Fraction 2	07OCT12:301SAL	0.23 U	2.2	0.00047 U	1.18 U
		Fraction 3	07OCT12:301SAL	0.23 U	1.9 U	0.00047 U	1.18 U
		Fraction 4	07OCT12:301SAL	0.23 U	1.9 U	0.00047 U	1.18 U
379GLBR- 121	Rep 1	Fraction 1	23OCT4:354SA2M	0.87	1.7	0.00047 U	0.60 U
		Fraction 2	23OCT4:354SA2M	0.22	0.8	0.00024 U	0.60 U
		Fraction 3	23OCT4:354SA2M	0.36	2.5	0.00024 U	1.00
		Fraction 4	23OCT4:354SA2M	0.36	2.5	0.00024 U	1.33
379GLBR- 121	Rep 2	Fraction 1	23OCT4:354SA2M	0.23 U	0.8	0.00047 U	0.60 U
		Fraction 2	23OCT4:354SA2M	0.23 U	0.8	0.00024 U	0.67
		Fraction 3	23OCT4:354SA2M	0.23	2.1	0.00024 U	1.00
		Fraction 4	23OCT4:354SA2M	0.23 U	2.1	0.00026	1.67
379GLBR- 121	Rep 3	Fraction 1	23OCT4:354SA2M	0.23 U	0.73 U	0.00024 U	0.60 U
		Fraction 2	23OCT4:354SA2M	0.23 U	1.3	0.00024 U	0.60 U
		Fraction 3	23OCT4:354SA2M	0.29	2.1	0.00024 U	2.00
		Fraction 4	23OCT4:354SA2M	0.29	1.3	0.00035	1.33
379GLBR- 154		Fraction 1	25OCT9:104SA3M	1.01	20.1	0.00149	0.72 U
		Fraction 2	25OCT9:104SA3M	0.64	14.2	0.00066	0.72 U
		Fraction 3	25OCT9:104SA3M	0.65	5.9	0.00255	3.82
		Fraction 4	25OCT9:104SA3M	0.65	5.9	0.00197	4.77
379GLBR- 460		Fraction 1	4DEC11:455SA2M	8.40	70.7	0.00156	7.23
		Fraction 2	4DEC11:455SA2M	6.70	35.8	0.00024 U	6.37
		Fraction 3	4DEC11:455SA2M	5.90	25.7	0.00076	2.31
		Fraction 4	4DEC11:455SA2M	9.80	22.3	0.00091	0.87
379GLBR- 461		Fraction 1	4DEC11:555SA2M	8.70	53.5	0.00066	8.39
		Fraction 2	4DEC11:555SA2M	6.80	34.5	0.00036	6.65
		Fraction 3	4DEC11:555SA2M	5.70	25.7	0.00107	4.05
		Fraction 4	4DEC11:555SA2M	7.90	20.2	0.00054	0.87
379GLBR- 464		Fraction 1	4DEC11:305SA3M	13.10	16.9	0.00024	6.91
379GLBR- 465		Fraction 1	4DEC11:375SA3M	11.20	13.2	0.00036	6.50

**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN LEACHATE SAMPLES  
(SBLT SEDIMENT EXTRACTIONS)**

MSL Code	Sponsor ID	Cr (ug/L) AA	Cu (ug/L) AA	Hg (ug/L) CVAF	Pb (ug/L) AA
<b>BIN A</b>					
379GLBR- 462	Fraction 1 4DEC12:055SA2M	8.00	56.4	0.00082	8.68
	Fraction 2 4DEC12:055SA2M	7.00	35.8	0.00040	8.10
	Fraction 3 4DEC12:055SA2M	8.20	27.4	0.00087	4.34
	Fraction 4 4DEC12:055SA2M	9.20	23.6	0.00098	1.16
379GLBR- 463	Fraction 1 4DEC11:155SA3M	9.50	10.5	0.00031	7.81
	Fraction 2 4DEC11:155SA3M	8.80	6.7	0.00030	5.50
	Fraction 3 4DEC11:155SA3M	12.50	5.5	0.00051	5.21
	Fraction 4 4DEC11:155SA3M	13.60	5.1	0.00045	4.34
<b>BIN B</b>					
379GLBR- 7	Fraction 1 07OCT12:571SBL	0.23 U	0.73 U	0.00047 U	0.60 U
	Fraction 2 07OCT12:571SBL	0.30	0.73 U	0.00047 U	0.60 U
	Fraction 3 07OCT12:571SBL	0.38	3.8	0.00024 U	3.81
	Fraction 4 07OCT12:571SBL	0.23	1.3	0.00039	0.69
379GLBR- 10	Fraction 1 07OCT1:071SBL	1.40	8.0	0.00047	9.83
	Fraction 2 07OCT1:071SBL	1.30	6.6	0.00024 U	9.17
	Fraction 3 07OCT1:071SBL	1.70	8.9	0.00024 U	10.81
	Fraction 4 07OCT1:071SBL	1.00	7.1	0.00025	9.17
379GLBR- 214	Fraction 1 31OCT9:304SB2M	0.23 U	2.4	0.00047 U	0.72 U
	Fraction 2 31OCT9:304SB2M	0.23 U	1.9 U	0.00047 U	0.72 U
	Fraction 3 31OCT9:304SB2M	0.23 U	1.9 U	0.00047 U	0.72 U
	Fraction 4 31OCT9:304SB2M	0.23 U	1.9 U	0.00055	0.72 U
379GLBR- 250	Fraction 1 31OCT2:404SB3M	0.60	4.7	0.00054	0.57 U
	Fraction 2 31OCT2:404SB3M	0.50	1.9	0.00064	0.62
	Fraction 3 31OCT2:404SB3M	0.70	5.2	0.00113	3.71
	Fraction 4 31OCT2:404SB3M	0.70	3.3	0.00143	3.09
379GLBR- 466	Fraction 1 4DEC12:505SB2M	15.40	15.5	0.00024 U	7.72
	Fraction 2 4DEC12:505SB2M	15.30	9.4	0.00024 U	6.50
	Fraction 3 4DEC12:505SB2M	14.60	8.5	0.00040	6.10
	Fraction 4 4DEC12:505SB2M	14.80	6.6	0.00030	4.06
379GLBR- 467	Fraction 1 4DEC1:005SB2M	10.00	17.9	0.00027	6.50
379GLBR- 468	Fraction 1 4DEC1:155SB2M	9.90	17.4	0.00027	6.10
379GLBR- 469	Fraction 1 4DEC1:255SB1/B3M	3.68	133.2	0.00047 U	10.98
	Fraction 2 4DEC1:255SB1/B3M	3.22	88.3	0.00047 U	5.66
	Fraction 3 4DEC1:255SB1/B3M	3.95	81.3	0.00047 U	1.77
	Fraction 4 4DEC1:255SB1/B3M	4.96	64.6	0.00047 U	0.72 U
379GLBR- 470	Fraction 1 4DEC1:305SB1/B3M	6.90	121.7	0.00133	18.70
379GLBR- 471	Fraction 1 4DEC1:355SB1/B3M	7.70	121.3	0.00132	10.57



**BUFFALO RIVER PILOT PROJECT (CF #379)  
METALS IN LEACHATE SAMPLES  
(SBLT SEDIMENT EXTRACTIONS)**

MSL Code	Sponsor ID	Cr (ug/L)	Cu (ug/L)	Hg (ug/L)	Pb (ug/L)
		AA	AA	CVAF	AA
BLANK-1	Fraction 1	0.23 U	1.9 U	0.00047	0.72 U
BLANK-1	Fraction 2	0.23 U	1.9 U	0.00047	0.72 U
BLANK-1	Fraction 3	0.23 U	1.9 U	0.00047 U	0.72 U
BLANK-1	Fraction 4	0.23 U	1.9 U	0.00047 U	0.72 U
BLANK-2	Fraction 1	0.30	0.73 U	0.00024 U	0.60 U
BLANK-2	Fraction 2	0.23 U	0.73 U	0.00024 U	0.60 U
BLANK-2	Fraction 3	0.30	0.80	0.00024 U	0.60 U
BLANK-2	Fraction 4	0.23 U	0.73 U	0.00032	0.60 U
BLANK-3	Fraction 1	0.20 U	0.90	0.00070	0.57 U
BLANK-3	Fraction 2	0.50	0.82 U	0.00028	0.57 U
BLANK-3	Fraction 3	0.20 U	0.82 U	0.00058	0.57 U
BLANK-3	Fraction 4	0.20 U	0.82 U	0.00026	0.57 U

**STANDARD REFERENCE MATERIAL**

1643c-1	Rep 1	19.59	27.5	NA	35.56
1643c-1	Rep 2	20.81	25.8	NA	36.48
1643c-2	Rep 1	19.50	26.5	NA	36.92
1643c-2	Rep 2	19.10	26.5	NA	37.85
1643c-3	Rep 1	19.80	26.3	NA	33.74
1643c-3	Rep 2	19.40	25.9	NA	36.95
1643c-4	Rep 1	NA	NA	NA	37.56
	certified value	19.00	22.3	NA	35.30
		±0.6	±2.8	NA	±0.90
1641b	Rep 1	NA	NA	1465	NA
1641b	Rep 2	NA	NA	1367	NA
1641b	Rep 3	NA	NA	1442	NA
1641b	Rep 4	NA	NA	1504	NA
1641b	Rep 5	NA	NA	1440	NA
1641b	Rep 6	NA	NA	1530	NA
1641b	Rep 7	NA	NA	1518	NA
	certified value	NA	NA	1520	NA
		NA	NA	±40	NA

**BUFFALO RIVER PILOT PROJECT (CF #379)**  
**METALS IN LEACHATE SAMPLES**  
**(SBLT SEDIMENT EXTRACTIONS)**

MSL Code	Sponsor ID	Cr (ug/L) AA	Cu (ug/L) AA	Hg (ug/L) CVAF	Pb (ug/L) AA
<b>REPLICATE ANALYSIS</b>					
379GLBR- 5	Rep 1 Fraction 1 07OCT12:381SAL	0.50	1.9 U	0.00094 U	0.72 U
379GLBR- 5	Rep 2 Fraction 1 07OCT12:381SAL	0.50	1.9 U	0.00094 U	1.66
379GLBR- 5	Rep 3 Fraction 1 07OCT12:381SAL	0.50	1.9 U	0.00047 U	1.24
	RSD %	0%	NA	NA	29%
379GLBR- 5	Rep 1 Fraction 2 07OCT12:381SAL	0.50	1.9 U	0.00047 U	0.72 U
379GLBR- 5	Rep 2 Fraction 2 07OCT12:381SAL	0.38	1.9 U	0.00047 U	0.83
379GLBR- 5	Rep 3 Fraction 2 07OCT12:381SAL	0.76	1.9 U	0.00047 U	1.66
	RSD %	35%	NA	NA	67%
379GLBR- 5	Rep 1 Fraction 3 07OCT12:381SAL	0.50	5.2	0.00060	6.63
379GLBR- 5	Rep 2 Fraction 3 07OCT12:381SAL	0.38	3.6	0.00069	5.80
379GLBR- 5	Rep 3 Fraction 3 07OCT12:381SAL	0.76	2.4	0.00047 U	4.56
	RSD %	35%	38%	14%	18%
379GLBR- 5	Rep 1 Fraction 4 07OCT12:381SAL	0.75	2.8	0.00047 U	4.56
379GLBR- 5	Rep 2 Fraction 4 07OCT12:381SAL	1.26	3.2	0.00047 U	3.31
379GLBR- 5	Rep 3 Fraction 4 07OCT12:381SAL	0.50	2.0	0.00047 U	2.07
	RSD %	46%	23%	NA	38%
379GLBR- 121	Rep 1 Fraction 1 23OCT4:354SA2M	0.87	1.7	0.00047 U	0.60 U
379GLBR- 121	Rep 2 Fraction 1 23OCT4:354SA2M	0.22	0.8	0.00047 U	0.60 U
379GLBR- 121	Rep 3 Fraction 1 23OCT4:354SA2M	0.36	0.73 U	0.00024 U	0.60 U
	RSD %	71%	72%	NA	NA
379GLBR- 121	Rep 1 Fraction 2 23OCT4:354SA2M	0.36	0.8	0.00024 U	0.60 U
379GLBR- 121	Rep 2 Fraction 2 23OCT4:354SA2M	0.23 U	0.8	0.00024 U	0.67
379GLBR- 121	Rep 3 Fraction 2 23OCT4:354SA2M	0.23 U	1.3	0.00024 U	0.60 U
	RSD %	NA	30%	NA	NA
379GLBR- 121	Rep 1 Fraction 3 23OCT4:354SA2M	0.23	2.5	0.00024 U	1.00
379GLBR- 121	Rep 2 Fraction 3 23OCT4:354SA2M	0.23 U	2.1	0.00024 U	1.00
379GLBR- 121	Rep 3 Fraction 3 23OCT4:354SA2M	0.23 U	2.1	0.00024 U	2.00
	RSD %	NA	10%	NA	43%
379GLBR- 121	Rep 1 Fraction 4 23OCT4:354SA2M	0.36	2.5	0.00024 U	1.33
379GLBR- 121	Rep 2 Fraction 4 23OCT4:354SA2M	0.23 U	2.1	0.00026	1.67
379GLBR- 121	Rep 3 Fraction 4 23OCT4:354SA2M	0.29	1.3	0.00035	1.33
	RSD %	22%	33%	30%	16%

U = Detected at or below detection limit.

NA = Not applicable/analyzed.

NS = Not spiked.

RSD % = Relative Standard Deviation.

**REPORT OF CHEMICAL ANALYSES**

**Volume 3: Organics**

**PROJECT: Buffalo River Pilot Project**

**Prepared for:**

**USEPA Great Lakes National Program Office  
Attn: Mr. Steve Garbaciak  
230 S. Dearborn  
Chicago, IL 60604**

REPORT OF: CHEMICAL ANALYSES

PROJECT: BUFFALO RIVER PILOT PROJECT

DATE: May 26, 1992

ISSUED TO: Mr. Steve Garbaciak  
Technical Project Manager  
USEPA Great Lakes National Program Office  
230 S. Dearborn  
Chicago, IL 60604

CF#: 379GLBR

## INTRODUCTION

This report summarizes the results from analyses performed on pilot study samples which were submitted by the U.S. Army Corps of Engineers Great Lakes Division, Buffalo District.

## SAMPLE CUSTODY

Samples were received in good condition from October 7, 1991 through December 5, 1991. Samples were logged in and stored as specified in the narrative. Samples were analyzed within the holding times specified in the QA plan. Any exceptions are noted in the narrative associated with each analysis.

## AIR SAMPLES

Five samples and one solvent blank were analyzed for dioxin/furans, PAH's and PCB's. Samples were stored at  $4\pm 2^{\circ}\text{C}$  prior to shipment to Twin City Testing for analysis of dioxin/furans, PAHs and PCBs. The samples were extracted in their entirety, therefore re-analysis was not possible. Included with this report are copies of the text which accompanied Twin City Testing's report for the air samples. Please refer to that data for information on extraction, analysis, quality control and any problems associated with analysis of the air samples. No samples were received to be archived.

## SAMPLE IDENTIFICATION

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
22OCT5:0011GA10	Air	D/F, PAH, PCB	379GLBR*112
23OCT4:4511GA20	Air	D/F, PAH, PCB	379GLBR*232
24OCT5:3011GA30	Air	D/F, PAH, PCB	379GLBR*233
25OCT1:4511GB10	Air	D/F, PAH, PCB	379GLBR*234
31OCT1:0011GB30	Air	D/F, PAH, PCB	379GLBR*272

## SEDIMENT and WATER SAMPLES

Fifty-one sediment samples were analyzed for PAHs and oil and grease. Forty-six sediment samples were analyzed for PAHs, PCBs and oil and grease. Samples were stored at  $-22\pm 3^{\circ}\text{C}$  prior to analysis. Oil and grease in sediment was determined according to "SOP for the Analysis of Solvent-Extractable Residue from Whole Sediment" taken from EPA-LLRS-GROSSE and supplied by the Great Lakes Large Lakes Lab. Twenty-three water samples were analyzed for PAHs, PCBs and oil and grease. Samples were stored at  $4\pm 2^{\circ}\text{C}$  prior to analysis. Since the organics lab extracted the entire sample for PAH and PCB analysis, and oil and grease used most of the duplicate sample, re-extractions were virtually impossible. On one occasion the organic samples were logged in incorrectly, as though each sample (including the duplicates) was a separate sample with each receiving an individual sample ID. In these

Instances, the organics analyst extracted each sample in its entirety, leaving no sample for oil and grease. To compensate for this error, metals samples or TOC samples collected from the same time and point of the processor were extracted for oil and grease. Although the metals and TOC samples had been preserved with acid, according to Standard Methods, acidifying an oil and grease sample does not affect the results. Oil and grease in water was determined following Standard Methods, 5520B, substituting methylene chloride for freon. Most water samples posed a problem using this method due to a high content of particulate matter in the sample. After some discussion with Eric Crecelius (program manager), the analyst was instructed to decant and extract the liquid layer. The particulate fraction was not extracted, but was archived for possible analysis in the future.

Water samples for PAHs and PCBs were extracted by shaking with methylene chloride in a large separatory funnel. Three consecutive extractions were performed on each sample, exchanging solvent after each extraction period following SOP #MSL-M-41. Samples were then cleaned using Silica/Alumina (5% deactivated) chromatography, followed by HPLC cleanup (Krahn et al. 1988). PAH extracts were analyzed using Gas Chromatography/Mass Spectrometry (GC/MS) in the selected ion mode (SIM). PCB extracts were analyzed using Gas Chromatography/Electron Capture Detection (GC/ECD). The column used was a J&W DB-5 capillary column (30m x 0.25mm I.D.). Sediment samples for PAHs and PCBs were extracted with methylene chloride using the ambient rolling technique. Three consecutive extractions were performed on each sample, exchanging solvent after each extraction period following SOP #MSL-M-42. Samples were then cleaned using Silica/Alumina (5% deactivated) chromatography followed by HPLC cleanup (Krahn et al. 1988). Extracts for PAHs were analyzed using Gas Chromatography/Mass Spectrometry (GC/MS) in the selected ion mode (SIM). Extracts for PCBs were analyzed using Gas Chromatography/Electron Capture Detection (GC/ECD). The column used was a J&W DB-5 capillary column (30m x 0.25mm I.D.).

For the majority of water samples to be analyzed for PAHs and PCBs, extraction exceeded the recommended 7-day holding time. However, due to the stable nature of PCB compounds and storage methods, the quality of the data should not be affected. Holding times from extraction to initial analyses were generally within EPA's recommended holding time of 40 days (EPA 1986). However, diluted samples were run approximately 30 to 40 days outside of these holding times. Values for diluted sample analyses generally agreed well with the initial quantitation, therefore, this added time does not appear to have biased the diluted results. Sediment samples for PAHs were extracted in 5 batches from 2/6/92 to 3/4/92 and analyzed from 3/31/92 to 4/18/92. Samples that required re-analysis were extracted on 4/22/92 and analyzed on 5/4/92. Samples that required dilution were analyzed on 5/1/92. One batch of sediment analyses requested as a rush by EPA GLNPO were extracted on 10/10/91 and analyzed on 10/14/92. Sediment samples for PCBs were extracted simultaneously with those for PAHs and analyzed from 4/20/92 through 4/26/92.

Target detection limits of 0.02 µg/L for PAHs in water were slightly exceeded in a number of cases. Detection limits ranged from 0.007 µg/L to 0.07 µg/L. In general, levels of PAHs exceeded these amounts in all but three samples. Target detection limits of 0.01 µg/L for PCBs in water were slightly exceeded for Aroclors. Detection limits averaged 0.05 µg/L to 0.2 µg/L for undiluted samples and from 0.5 to 2 µg/L for diluted samples. In most cases, PCBs in the samples analyzed exceeded these amounts. Sediment PAH target detection limits of 0.02 µg/Kg were slightly exceeded, ranging from 0.004 to 0.053 µg/Kg. Sediment PCB target detection limits of 0.02 µg/Kg were slightly exceeded, ranging from 0.025 to 0.060 µg/Kg. Detection limits reported were instrument detection limits based on a minimum area, background noise and the analyst's judgement on the level that was quantifiable.

**SAMPLE IDENTIFICATION**

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
07OCT12:341SAO	Sediment	PAH, Oil & Gr	379GLBR*13
07OCT11:101SBO	Sediment	PAH, Oil & Gr	379GLBR*14
07OCT12:401SAO	Sediment	PAH, Oil & Gr	379GLBR*16
07OCT1:331SCO	Sediment	PAH, Oil & Gr	379GLBR*18
07OCT2:041SDO	Sediment	PAH, Oil & Gr	379GLBR*19
07OCT1:441SCO	Sediment	PAH, Oil & Gr	379GLBR*20
07OCT1:011SBO	Sediment	PAH, Oil & Gr	379GLBR*23
07OCT2:121SDO	Sediment	PAH, Oil & Gr	379GLBR*24
08OCT5:402SD1O	Sediment	PAH, Oil & Gr	379GLBR*51
08OCT5:302SD2O	Sediment	PAH, Oil & Gr	379GLBR*53
09OCT11:502SA3O	Sediment	PAH, Oil & Gr	379GLBR*57
09OCT11:282SA1O	Sediment	PAH, Oil & Gr	379GLBR*59
09OCT10:152SB2O	Sediment	PAH, Oil & Gr	379GLBR*66
09OCT10:352SB3O	Sediment	PAH, Oil & Gr	379GLBR*67
09OCT4:352SC2O	Sediment	PAH, Oil & Gr	379GLBR*74
09OCT4:162SC3O	Sediment	PAH, Oil & Gr	379GLBR*75
22OCT5:563SA2O	Sediment	PAH, Oil & Gr	379GLBR*111
23OCT1:303SA2O	Sediment	PAH, Oil & Gr	379GLBR*113
23OCT4:284SA2O	Sediment	PAH, Oil & Gr	379GLBR*122
23OCT4:294SA2O	Sediment	PAH, Oil & Gr	379GLBR*124
24OCT2:203SA3O	Sediment	PAH, Oil & Gr	379GLBR*141
24OCT5:503SA3O	Sediment	PAH, Oil & Gr	379GLBR*143
25OCT9:224SA3O	Sediment	PAH, Oil & Gr	379GLBR*151
25OCT9:224SA3O	Sediment	PAH, Oil & Gr	379GLBR*152
25OCT2:153SB1O	Sediment	PAH, Oil & Gr	379GLBR*176
25OCT2:294SB1O	Sediment	PAH, Oil & Gr	379GLBR*182
25OCT2:284SB1O	Sediment	PAH, Oil & Gr	379GLBR*183
30OCT2:493SB2O	Sediment	PAH, Oil & Gr	379GLBR*198
30OCT4:413SB2O	Sediment	PAH, Oil & Gr	379GLBR*206
31OCT9:264SB2O	Sediment	PAH, Oil & Gr	379GLBR*220
31OCT9:254SB2O	Sediment	PAH, Oil & Gr	379GLBR*221
31OCT10:453SB3O	Sediment	PAH, Oil & Gr	379GLBR*242
31OCT2:303SB3O	Sediment	PAH, Oil & Gr	379GLBR*244
31OCT2:384SB3O	Sediment	PAH, Oil & Gr	379GLBR*247
31OCT2:364SB3O	Sediment	PAH, Oil & Gr	379GLBR*249
18NOV2:553SC1O	Sediment	PAH, Oil & Gr	379GLBR*280
18NOV4:403SC1O	Sediment	PAH, Oil & Gr	379GLBR*284
19NOV9:004SC1O	Sediment	PAH, Oil & Gr	379GLBR*303
19NOV9:014SC1O	Sediment	PAH, Oil & Gr	379GLBR*304
19NOV2:203SC2O	Sediment	PAH, Oil & Gr	379GLBR*313
19NOV3:553SC2O	Sediment	PAH, Oil & Gr	379GLBR*315
19NOV4:504SC2O	Sediment	PAH, Oil & Gr	379GLBR*324
19NOV4:504SC2O	Sediment	PAH, Oil & Gr	379GLBR*325
20NOV1:003SC3O	Sediment	PAH, Oil & Gr	379GLBR*342
20NOV1:553SC3O	Sediment	PAH, Oil & Gr	379GLBR*344
20NOV3:154SC3O	Sediment	PAH, Oil & Gr	379GLBR*351
20NOV3:154SC3O	Sediment	PAH, Oil & Gr	379GLBR*352

21NOV12:003SD1O	Sediment	PAH, Oil & Gr	379GLBR*376
21NOV2:433SD1O	Sediment	PAH, Oil & Gr	379GLBR*380
21NOV5:224SD1O	Sediment	PAH, Oil & Gr	379GLBR*396
21NOV5:224SD1O	Sediment	PAH, Oil & Gr	379GLBR*397
07OCT12:11SCO	Sediment	PAH, PCB, Oil & Gr	379GLBR*15
07OCT12:091SAO	Sediment	PAH, PCB, Oil & Gr	379GLBR*17
07OCT12:531SBO	Sediment	PAH, PCB, Oil & Gr	379GLBR*21
07OCT1:521SDO	Sediment	PAH, PCB, Oil & Gr	379GLBR*22
08OCT5:132SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*46
09OCT11:382SA2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*58
09OCT9:542SB1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*65
09OCT4:562SC1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*73
22OCT11:317SA1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*104
22OCT11:378SA1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*107
22OCT4:203SA2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*108
23OCT4:274SA2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*123
24OCT10:457SA2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*136
24OCT10:508SA2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*139
24OCT6:203SA3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*145
25OCT9:214SA3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*153
25OCT9:307SA3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*166
25OCT9:368SA3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*167
25OCT12:153SB1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*174
25OCT2:254SB1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*181
25OCT3:527SB1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*193
25OCT3:578SB1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*196
30OCT2:573SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*199
31OCT9:027SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*210
31OCT9:078SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*213
31OCT9:244SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*222
31OCT11:503SB3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*243
31OCT2:354SB3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*248
1NOV10:208SB3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*268
1NOV10:157SB3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*269
18NOV3:503SC1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*282
19NOV9:347SC1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*288
19NOV8:594SC1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*302
19NOV9:238SC1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*307
19NOV11:303SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*311
19NOV4:504SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*323
20NOV8:157SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*337
20NOV11:443SC3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*340
20NOV3:154SC3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*350
20NOV3:337SC3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*357
20NOV3:278SC3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*360
21NOV1:353SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*378
21NOV5:078SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*384
21NOV5:107SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*388
21NOV5:224SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*395
23OCT4:459LA2O	Oil	PAH, PCB, Oil & Gr	379GLBR*128

23OCT5:2010LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*132
25OCT10:1810LA3O	Water	PAH, PCB	379GLBR*160*
25OCT10:1910LA3O	Water	PAH, PCB	379GLBR*161*
25OCT10:2010LA3M	Water	Oil & Gr (metals)	379GLBR*163**
25OCT9:549LA3O	Oil	PAH, PCB	379GLBR*164*
25OCT10:009LA3C	Oil	Oil & Gr (TOC)	379GLBR*165**
25OCT3:0810LB1O	Water	PAH, PCB, Oil & Gr	379GLBR*187
30OCT4:206LA3O	Water	PAH, PCB, Oil & Gr	379GLBR*204
31OCT10:119LB2O	Oil	PAH, PCB, Oil & Gr	379GLBR*226
31OCT10:2310LB2O	Water	PAH, PCB, Oil & Gr	379GLBR*227
31OCT3:119LB3O	Oil	PAH, PCB	379GLBR*252*
31OCT3:109LB3O	Oil	PAH, PCB	379GLBR*253*
31OCT2:5710LB3M	Water	Oil & Gr (metals)	379GLBR*254**
31OCT3:069LB3M	Oil	Oil & Gr (metals)	379GLBR*255**
31OCT3:0110LB3O	Water	PAH, PCB	379GLBR*256*
31OCT3:0010LB3O	Water	PAH, PCB, Oil & Gr	379GLBR*257
6NOV12:003LO	Water	PAH, PCB, Oil & Gr	379GLBR*275
18NOV5:559LC1O	Oil	PAH, PCB, Oil & Gr	379GLBR*286
18NOV6:1510LC1O	Water	PAH, PCB, Oil & Gr	379GLBR*287*
19NOV5:519LC2O	Oil	PAH, PCB, Oil & Gr	379GLBR*326
19NOV5:0510LC2O	Water	PAH, PCB, Oil & Gr	379GLBR*331
20NOV2:193LC3O	Water	PAH, PCB, Oil & Gr	379GLBR*361
20NOV2:419LC3O	Oil	PAH, PCB, Oil & Gr	379GLBR*366
20NOV3:0110LC3O	Water	PAH, PCB, Oil & Gr	379GLBR*370
21NOV3:2610LD1O	Water	PAH, PCB, Oil & Gr	379GLBR*398
21NOV4:169LD1O	Oil	PAH, PCB, Oil & Gr	379GLBR*404

\*Samples extracted for PAH and PCB only.

\*\*Samples extracted for oil/grease and metals or TOC.

\*Sample was not analyzed.

#### ARCHIVED SAMPLES

<u>Sponsor ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>Battelle ID</u>
21OCT12:483SA1O	Sediment	PAH, Oil & Gr	379GLBR*83
21OCT12:493SA1O	Sediment	PAH, Oil & Gr	379GLBR*84
21OCT5:444SA1O	Sediment	PAH, Oil & Gr	379GLBR*92
21OCT5:464SA1O	Sediment	PAH, Oil & Gr	379GLBR*93
22NOV12:153SD2O	Sediment	PAH, Oil & Gr	379GLBR*408
22NOV1:153SD2O	Sediment	PAH, Oil & Gr	379GLBR*409
22NOV3:104SD2O	Sediment	PAH, Oil & Gr	379GLBR*418
22NOV3:104SD2O	Sediment	PAH, Oil & Gr	379GLBR*419
25NOV1:003SD3O	Sediment	PAH, Oil & Gr	379GLBR*436
25NOV1:403SD3O	Sediment	PAH, Oil & Gr	379GLBR*438
25NOV4:004SD3O	Sediment	PAH, Oil & Gr	379GLBR*444
25NOV4:004SD3O	Sediment	PCB, Oil & Gr	379GLBR*450
08OCT5:102SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*46-T1
08OCT5:112SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*46-T2
08OCT5:122SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*46-T3
08OCT5:132SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*46-M1
21OCT12:473SA1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*82



21OCT5:454SA1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*91
30OCT2:473SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*199-T1
30OCT4:013SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*199-T2
30OCT2:523SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*199-M1
30OCT4:453SB2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*199-M2
19NOV4:504SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*323-T1
19NOV4:504SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*323-T2
19NOV4:504SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*323-M1
19NOV4:504SC2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*323-M2
20NOV2:193LC3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*361-1
21NOV5:078SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*384-T1
21NOV5:078SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*384-T2
21NOV5:078SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*384-M1
21NOV5:078SD1O	Sediment	PAH, PCB, Oil & Gr	379GLBR*384-M2
22NOV1:453SD2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*412
22NOV3:104SD2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*417
22NOV3:308SD2O	Sediment	PAH, PCB, Oil & Gr	379GLBR*423
25NOV2:203SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*440
25NOV4:004SD3O	Sediment	PAH, PCB, Oil & Gr	379GLBR*449
21OCT6:1810LA1O	Water	PAH, PCB, Oil & Gr	379GLBR*97
21OCT6:2010LA1O	Water	PAH, PCB, Oil & Gr	379GLBR*97-T1
21OCT6:2210LA1O	Water	PAH, PCB, Oil & Gr	379GLBR*97-T2
21OCT6:1610LA1O	Water	PAH, PCB, Oil & Gr	379GLBR*97-T3
23OCT4:459LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*128-1
23OCT4:459LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*128-2
23OCT4:459LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*128-3
23OCT5:2010LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*132-1
23OCT5:2010LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*132-2
23OCT5:2010LA2O	Water	PAH, PCB, Oil & Gr	379GLBR*132-3
25OCT3:0710LB1O	Water	PAH, PCB, Oil & Gr	379GLBR*187-1
30OCT4:206LA3O	Water	PAH, PCB, Oil & Gr	379GLBR*204-1
31OCT10:129LB2O	Oil	PAH, PCB, Oil & Gr	379GLBR*226-1
31OCT10:2110LB2O	Water	PAH, PCB, Oil & Gr	379GLBR*227-1
6NOV12:003LO	Water	PAH, PCB, Oil & Gr	379GLBR*275-1
18NOV5:559LC1O	Oil	PAH, PCB, Oil & Gr	379GLBR*286-1
18NOV5:559LC1O	Oil	PAH, PCB, Oil & Gr	379GLBR*286-T1
18NOV5:559LC1O	Oil	PAH, PCB, Oil & Gr	379GLBR*286-T2
18NOV5:559LC1O	Oil	PAH, PCB, Oil & Gr	379GLBR*286-M1
18NOV5:559LC1O	Oil	PAH, PCB, Oil & Gr	379GLBR*286-M2
18NOV6:1510LC1O	Water	PAH, PCB, Oil & Gr	379GLBR*287-1
19NOV5:519LC2O	Oil	PAH, PCB, Oil & Gr	379GLBR*326-1
19NOV5:0510LC2O	Water	PAH, PCB, Oil & Gr	379GLBR*331-T1
19NOV5:0510LC2O	Water	PAH, PCB, Oil & Gr	379GLBR*331-T2
19NOV5:0510LC2O	Water	PAH, PCB, Oil & Gr	379GLBR*331-M1
19NOV5:0510LC2O	Water	PAH, PCB, Oil & Gr	379GLBR*331-M2
20NOV2:419LC3O	Oil	PAH, PCB, Oil & Gr	379GLBR*366-1
20NOV3:0110LC3O	Water	PAH, PCB, Oil & Gr	379GLBR*370-1
21NOV3:2610LD1O	Water	PAH, PCB, Oil & Gr	379GLBR*398-1
21NOV4:169LD1O	Oil	PAH, PCB, Oil & Gr	379GLBR*404-1

21NOV4:169LD1O	Oil	PAH, PCB, Oil & Gr	379GLBR*404-T1
21NOV4:169LD1O	Oil	PAH, PCB, Oil & Gr	379GLBR*404-T2
21NOV4:169LD1O	Oil	PAH, PCB, Oil & Gr	379GLBR*404-M1
21NOV4:169LD1O	Oil	PAH, PCB, Oil & Gr	379GLBR*404-M2
22NOV2:3010LD2O	Water	PAH, PCB, Oil & Gr	379GLBR*426
22NOV2:3010LD2O	Water	PAH, PCB, Oil & Gr	379GLBR*426-1
22NOV2:539LD2O	Oil	PAH, PCB, Oil & Gr	379GLBR*431
22NOV2:539LD2O	Oil	PAH, PCB, Oil & Gr	379GLBR*431-1
25NOV4:0010LD3O	Water	PAH, PCB, Oil & Gr	379GLBR*452
25NOV4:0010LD3O	Water	PAH, PCB, Oil & Gr	379GLBR*452-1
25NOV4:009LD3O	Oil	PAH, PCB, Oil & Gr	379GLBR*453
25NOV4:009LD3O	Oil	PAH, PCB, Oil & Gr	379GLBR*453-1

## OIL AND GREASE

Analysis of oil and grease in water following the SOP which we received from the Large Lakes Lab was not possible. Most of the samples had a very high content of oil and fine sediment. When the sample was shaken, only one phase was apparent instead of two as described in the method. Consequently, after discussion with Eric Crecelius (program manager), the analyst allowed the sediment to settle, decanted and analyzed the liquid, and archived the sediment. Some sediment samples analyzed for oil and grease were rerun due to the formation of a drying agent residue in the extract which caused higher values. In some cases, ash passed through the filter into the extract, which caused higher values. In other cases, the replication between field replicates was not in good agreement. We wanted to ensure that the poor agreement was due to some artifact other than the methodology. If the reruns differed substantially from the original value obtained, the rerun value was reported. If the differences were slight, the original value was reported. All samples were analyzed outside the standard EPA holding time of 28 days. This was due in part to the addition of this analysis to the sampling scheme after the program was underway and partially to sample backlog in the organics laboratory. The quality of the data should not be affected since the sediments were stored at  $-22^{\circ}\pm 5^{\circ}\text{C}$  and the water samples were stored at  $4^{\circ}\pm 2^{\circ}\text{C}$  prior to analysis. Matrix spikes for sediments were within the criteria outlined in the QAPP for organic samples, except for samples 379GLBR\*182 and 384 which were treated sediment. Recoveries were higher in those samples probably due to matrix interference.

## PAHS AND PCBS

### Water

Matrix problems were evident in these samples due to high concentrations of oil. Many spike and surrogate recoveries were outside of the criteria. EPA GLNPO (Rick Fox) indicated they wanted the entire sample extracted, including the oil adhered to the sides of the bottles. When the analysts extracted the samples, they found that the methylene chloride formed an emulsion with the oily water which could not be separated out. This caused a problem in the clean-up step and apparently caused some matrix interference in the samples. Evidence for this assumption comes from spike blank recoveries, replicate RSDs and blank data, which were within the established ARCS criteria.

One method blank was extracted and analyzed for PAHs with the samples. Naphthalene and Benzo(ghi)perylene were detected at levels near detection limits in the blank. These levels were less than five times the levels found in all but three samples. The corresponding sample values for these samples were flagged with a "B" to indicate a possible bias due to blank contamination. One method

blank was extracted and analyzed for PCB Aroclors with the samples. No PCBs were detected in the method blanks.

Three stable isotopically labelled PAH compounds were added as surrogates prior to extraction to assess the efficiency of the analysis. The majority of recoveries for all three surrogates were generally below the lower QC limits (40-120%). This is mainly due to very high PAH concentrations and oily material associated with these samples. Performing an efficient extraction on these samples was very difficult. Samples with little or no PAHs had much higher surrogate recoveries, including the method blank. Due to limited sample volumes, holding times and extremely difficult sample matrices, no re-extractions were performed. Overall, PAH levels may be somewhat biased low based on the surrogate recoveries. However, the values for some of the higher weight PAH compounds are nearing their solubility limit in water and the levels quantified are more likely a function of the amount of oily material in the sample rather than a truly dissolved quantity. Two compounds, tetrachlorometaxylene (TCMX) and octachloronaphthalene (OCN) were added as surrogates to all samples prior to extraction of PCBs to assess the efficiency of the analysis. Recoveries of four samples for TCMX and OCN slightly exceeded the QC guidelines of 40-120%. All recoveries were above 20%, therefore no re-extractions were performed.

One sample was spiked in duplicate with a PAH standard (see MSL-M-42). However, the level spiked was generally five to ten times lower than the amount in the native sample. This precluded accurate determination of spike recoveries for all but one compound, benzo(k)fluoranthene, which was not present above detection limits in the native sample. Recoveries for PAHs were generally poor due to matrix interference and non-homogenous samples. In addition, spike concentrations were generally lower than the sample concentration, therefore were difficult to detect. For PCBs, one sample was spiked in duplicate with Aroclor 1254. Unfortunately, it appears that one of the two samples was not spiked after all, therefore matrix spike recoveries were only reported for one of the two samples. Aroclor 1254 spike recovery was 112% for this sample.

One sample was extracted in triplicate to assess the precision of PAH analyses. RSD values ranged from 4 to 40%. The majority of RSD values were between 20-30%, which exceeds the precision goal of 20%. These values indicate relatively good precision, considering high levels of PAHs in the samples and the difficulty of performing representative extractions on these oily, complex matrices. One sample was extracted in triplicate to assess the precision of PCB analyses. The sample analyzed contained high levels of Aroclor 1248 and needed dilution prior to quantification. The RSD value for the triplicate analyses was 41%. This exceeded the QAPP precision goal of 20%. Some error was introduced due to dilution which contributed to the higher RSD value. In addition, these water samples contained high levels of PCBs and oily material which are difficult to extract entirely from the samples. Some variability may come from non-homogenous field replicates. The combination of these factors most likely contributed to high RSD values.

Standard reference materials (SRMs) are not available for organics in water samples.

### Sediments

Matrix problems were evident in these samples due to high concentrations of oil. Many spike and surrogate recoveries were outside of the criteria. When the analysts extracted the samples, they found that the methylene chloride formed an emulsion with the oily water content of the sediments which could not be separated out. This caused a problem in the clean-up step and apparently caused some matrix interference in the samples. Evidence for this assumption comes from spike blank recoveries, replicate RSDs and blank data, which were within the established ARCS criteria.

Seven method blanks, one with each batch, were extracted and analyzed for PAHs. Naphthalene and Phenanthrene were detected at levels near detection limits. Sample concentrations were five times these levels in all samples but three. One method blank was extracted with each batch for PCBs. No PCBs were detected in the blanks.

Our stable isotopically labelled PAH compounds were added as surrogates prior to extraction to assess the efficiency of the analyses. Recoveries of two of the surrogates d8 naphthalene and d10 acenaphthalene were occasionally low (<40%) due to their volatile nature. The surrogate d14 Dibenzo (a,h) Anthracene was detected both above and below the acceptable limit. The lower values indicate that this particular surrogate may be susceptible to degradation. We are presently replacing d14 dibenzo (a,h) anthracene with d10 pyrene in an effort to achieve better recoveries. Two compounds, tetrachlorometaxylene (TCMX) and octachloronaphthalene (OCN) were added to all samples prior to extraction for PCBs to assess the efficiency of the analyses. Recoveries for TCMX were within the QC guidelines of 40-120%, except for one sample. OCN recoveries were generally high due to underspiking, which caused difficulty in quantification of this surrogate.

Five samples and three blanks were spiked in duplicate with a PAH combined stock standard. Most samples contained much higher concentrations of PAHs than were spiked, therefore spike recoveries were usually outside the required criteria (40-120%). Matrix spikes with poor recoveries in which the spike concentration was approximately the same as the sample or higher, are probably a result of matrix interference. Matrix spikes for sample 379GLBR\*46 were not reported because the sample results were suspect from the first analysis, the sample was rerun producing very different results, but the spikes were not rerun. Spike data for the initial analysis of sample 379GLBR\*111 were not reported as the results were questionable and the sample and spikes were re-analyzed. The reruns were reported. Two samples and two method blanks were spiked in duplicate with Aroclor 1254 for PCBs. Several recoveries were slightly above the acceptable limit (40-120%). One of the blank spike recoveries was abnormally high, possibly due to an accidental duplicate spike on that sample.

Five samples were extracted in triplicate for PAHs to assess the precision of the analyses. Precision was measured by calculating the relative standard deviation (RSD) between the replicate results for each of 17 compounds. RSD values ranged from 3 to 25% for with one RSD of 52%. The majority of the RSD values were between 10 and 20%, indicating good precision. Two samples were extracted in triplicate for PCBs to assess the precision of the analyses. Sample 379GLBR\*199 did not replicate well with an RSD of 89% for the Aroclors detected, possibly due to uneven oil distribution within the sediment matrix.

The SRM generally used for sediments (NIST 1941) is not certified for PCB Aroclors or PAHs, only individual congeners. One other SRM is available and is certified for PCB Aroclor 1254, (HS-2 from NRCC), however, it was originally certified using a packed column. Our experience has shown that there is little or no 1254 pattern when capillary column GC is used. Therefore, we do not use it as a SRM for Aroclors. SRM data is reported for PAHs and compared using RSD values.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Polynuclear Aromatic Hydrocarbons (PAHs)  
LABORATORY: Twin City Testing, Inc.  
MATRIX: Air  
SAMPLE NUMBER: 6

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
8270	40-120%	±20%	N/A

HOLDING TIMES Not specified.

DETECTION LIMITS Detection limits were different for each sample and each compound. Detection limits ranged from 0.75 to 3.8 µg/sample. Please refer to the summary table for specific detection limits.

METHOD BLANKS A laboratory method blank was prepared and analyzed with each sample extraction batch. The blanks were free of the target PAH analytes, with the exception of a trace background level (77 nanograms) of naphthalene. The naphthalene levels determined for the affected samples were all higher than the naphthalene level in the blank by 20 times or more. This indicates that the sample processing steps did not contribute significantly to the levels reported for the samples.

MATRIX SPIKES The amount of material was insufficient to prepare matrix spikes. Two laboratory spike samples were prepared with the air sample batch. Spike recoveries ranged from 52-132%, which are all within the 50-135% target ranges designated for these analyses.

REPLICATES The amount of material was insufficient for the preparation of replicates.

SRMS SRMs are not available for PAHs in air samples.

SURROGATES The designated range for the surrogate compounds in the PAH samples is 50-150% for at least two of the three surrogates in each sample. All the surrogate recoveries in the PAH samples fell within this range, with the exception of perdeuterated pyrene (227%) in sample 379GLBR\*112ABE. Since the remaining two surrogates in this sample were recovered at acceptable levels, the recovery criteria were met and no corrective action was required. The raw area count for the perdeuterated naphthalene internal standard in sample 379GLBR\*272ABD was 269% of the value for the corresponding standard in the daily continuing calibration analysis. The target range for internal standard areas in the sample extracts is 50-200% of the daily continuing calibration values. Upon dilution of this sample extract, the naphthalene internal standard area fell into the acceptable range.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Polychlorinated biphenyls (PCBs)  
LABORATORY: Twin City Testing, Inc.  
MATRIX: Air  
SAMPLE NUMBER: 6

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
680	40-120%	±20%	N/A

HOLDING TIMES Not specified.

DETECTION LIMITS Detection limits were different for each sample and each compound. Detection limits ranged from 60 to 120 ng/sample. Please refer to the summary table for specific detection limits.

METHOD BLANKS A laboratory method blank was prepared and analyzed with each sample extraction batch. The blanks were free of the target PCB analytes. A low recovery was achieved for the 2-Fluorobiphenyl surrogate in the PCB blank due to inadvertent volatilization of this compound during the concentration steps. Since the entire sample was consumed in the extraction process, any reprocessing was not possible. The blank associated with the impinger samples exhibited a low recovery (35%) for the perdeuterated anthracene surrogate. Since good recoveries were achieved for the other two surrogates in this blank, no corrective action was required.

MATRIX SPIKES The amount of material was insufficient to prepare matrix spikes. Two laboratory spike samples were prepared with each batch. Spiked native compounds were recovered at levels ranging from 52-132%, which are all within the 50-135% target ranges designated for these analytes.

REPLICATES The amount of material was insufficient for preparation of replicates.

SRMS SRMs are not available for PCBs in air samples.

SURROGATES The recoveries of the surrogate compounds fell within the 50-125% guidelines specified in Method 680, with the exception of the 2-Fluorobiphenyl recovery (47%) in sample 379GLBR\*112F.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Dioxins/furans  
LABORATORY: Twin City Testing, Inc.  
MATRIX: Air  
SAMPLE NUMBER: 6

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
EPA Method 23	Not specified	Not specified	Not specified

HOLDING TIMES Not specified.

DETECTION LIMITS Detection limits were different for each sample and each compound. Generally, they varied from 0.013 to 0.140 ng/sample. Please refer to the summary table for specific detection limits.

METHOD BLANKS One laboratory method blank was prepared and analyzed with the sample extraction batch. The blanks were free of dioxins and furans, with the exception of trace background levels of PeCDD (19 picograms), HpCDD (6.3 picograms) and OCDD (92 picograms). The levels determined for the affected isomers in the actual samples were higher than the corresponding blank levels by 3-100 times. It should be noted, however, that levels less than five times higher than the background are not generally considered statistically different from the background.

MATRIX SPIKES The amount of sample material was insufficient to prepare matrix spikes. Two quality control dioxin/furan spike samples were prepared with the sample batch. The data show that the spiked native compounds were recovered at levels ranging typically from 87-130%. The only compound with recoveries outside of this range was 1,2,3,4,7,8,9-HpCDF, which exhibited a recovery of 160% in each of the spike samples. It should be noted, that quality control ranges for native spike sample recoveries are not specified in the method.

REPLICATES The amount of sample material was insufficient for the preparation of replicates.

SRMS SRMS are not available for dioxin/furans in air samples.

SURROGATES The recoveries of the isotopically-labeled dioxin/furan internal and surrogate standards generally ranged from 70-130%. All the recoveries were within the target ranges specified in the method, with the exceptions of the labeled 1,2,3,7,8-PeCDD in samples 379GLBR\*112F (37%), 379GLBR\*272ABD (37%), and the lab spike duplicate (36%). Slightly elevated recoveries were obtained for selected surrogate compounds in samples 379GLBR\*233ABD (labeled 1,2,3,4,7,8-HxCDD, 145%), 379GLBR\*272ABD (labeled 1,2,3,4,7,8-HxCDD, 148%), the laboratory spike (labeled 1,2,3,4,7,8,9-HpCDF, 135%), and the laboratory spike duplicate (labeled 1,2,3,4,7,8,9-HpCDF, 135%). The native 1,2,3,7,8-PeCDD concentrations should be accurate for these samples since

quantitation is based on isotope dilution. The native 1,2,3,4,7,8-HxCDD and 1,2,3,4,7,8,9-HpCDF concentrations in these four samples may, however, be slightly elevated since both the native and surrogate isomers are compared to a separate labeled isomer.



## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Oil and grease  
LABORATORY: Battelle Marine Science Laboratories  
MATRIX: Water  
SAMPLE NUMBER: 23

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
5520B	70-130%	±20%	Not specified

HOLDING TIMES Not specified.

DETECTION LIMITS The detection limit for this method was determined to be 1.1 mg/L.

METHOD BLANKS Eight method blanks were prepared and analyzed, at least one with each batch extracted. Oil and grease was less than the detection limit in all blanks.

MATRIX SPIKES Not required by the QAPP. Two samples were spiked in duplicate with a known concentration of pump oil. Recoveries ranged from 95 to 134%. Laboratory spike blanks (not required) were prepared and analyzed with recoveries ranging from 76 to 78%.

REPLICATES Two samples were extracted and analyzed in triplicate. Relative standard deviations were 3 and 17%.

SRMS SRMs are not available for oil and grease.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Oil and grease  
LABORATORY: Battelle Marine Science Laboratories  
MATRIX: Sediment  
SAMPLE NUMBER: 97

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
EPA-LLRS-GROSSE	70-130%	+20%	N/A

HOLDING TIME Not specified.

DETECTION LIMIT The detection limit was determined as 630 µg/g.

METHOD BLANKS Nine blanks were prepared and analyzed; at least one with each batch. Oil and grease was not detected in any of the blanks.

MATRIX SPIKES Not required in the QAPP. Five matrix spikes in duplicate were spiked with pump oil, one with each batch. All recoveries were within 70-130%, with the exception of 379GLBR\*182 and 379GLBR\*384, where recoveries ranged from 142 to 158%. These two samples were ash material which possibly caused some matrix interference in this method. Laboratory blank spikes were prepared and analyzed, with recoveries ranging from 87 to 115%.

REPLICATES Five samples were extracted and analyzed in triplicate; at least one per batch. All triplicates were within the range of precision, with the exception of 379GLBR\*111 and 379GLBR\*384. Both samples replicated with a RSD of 22%, possibly caused by a residue from dissolution of sodium sulfate (drying agent) when mixed with the sediment that passed through the filtration step into the extract.

SRMS SRMs are not available for oil and grease.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Polynuclear Aromatic Hydrocarbons (PAHs)  
LABORATORY: Battelle Marine Science Laboratories  
MATRIX: Water  
SAMPLE NUMBER: 23

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
MSL-M-41	40-120%	+20%	0.02µg/L

**HOLDING TIME** Holding times for extraction were exceeded for all samples by approximately 60 days. However, all samples were stored at 4°C preventing biodegradation of the samples. All samples were analyzed within 40 days of extraction, which meets the established criteria.

**DETECTION LIMITS** Detection limits ranged from 0.007 to 0.07 µg/L. The higher detection limits were associated with samples requiring dilution or those with high PAH concentrations that required smaller extraction volumes.

**METHOD BLANKS** One method blank was analyzed with the samples. The blank was free of PAH compounds except Naphthalene and benzo(ghi)perylene were detected at levels near detection limits in the blank. These levels were less than five times the levels found in all but three samples. Surrogate recoveries in the blanks ranged from 61 to 67%.

**MATRIX SPIKES** One sample was spiked in duplicate for PAHs. The matrix spike recoveries were all negative percentages except for Benzo(a)anthracene (2026%), benzo(k)fluoranthene (84%) and dibenzo(a,h)anthracene (4%). The concentration of the spike was generally much lower than the sample concentration, causing difficulty in detecting the spike signal. The spike duplicate showed recoveries ranging from 3 to 197% with a negative recovery for benzo(b)fluoranthene. This indicates some matrix interference.

**REPLICATES** One sample was extracted and analyzed in triplicate. Relative standard deviations were generally between 25 and 35%. Dibenzo(a,h)anthracene RSD was 47%. Naphthalene, acenaphthalene and acenaphthene had RSDs below 20%, which is within the established criteria.

**SRMS** SRMs are not available for PAHs in water.

**SURROGATES** Most surrogates were outside the established criteria, probably due to matrix interference and high sample concentrations relative to surrogate concentrations used.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Polychlorinated biphenyls (PCBs)  
LABORATORY: Battelle Marine Science Laboratories  
MATRIX: Water  
SAMPLE NUMBER: 23

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
MSL-M-41	40-120%	+20%	0.01µg/L

**HOLDING TIME** Holding times for extraction were exceeded for all samples by approximately 60 days. However, all samples were stored at 4°C preventing biodegradation of the samples. All samples were analyzed within 40 days of extraction, which meets the established criteria.

**DETECTION LIMITS** The detection limits ranged from 0.05µg/L to 0.2 µg/L for undiluted samples and from 0.5µg/L to 2µg/L for diluted samples.

**METHOD BLANK** One method blank was prepared and analyzed with the samples. No PCBs were detected in the blank. Surrogate recoveries were 62 and 96% in the blank.

**MATRIX SPIKE** One matrix spike was prepared and analyzed with the samples for Aroclor 1254. The recovery was 112% and the surrogate recoveries were 44 and 50%. A duplicate spike was set up but was not spiked.

**REPLICATES** One sample was extracted and analyzed in triplicate with an RSD of 41%.

**SURROGATES** Most samples were within the criteria for surrogate recovery of 40 to 120%. Seven of 23 samples had one surrogate recovery outside the required criteria. Both surrogate recoveries for 1 sample were below the required criteria.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Polynuclear Aromatic Hydrocarbons (PAHs)  
LABORATORY: Battelle Marine Science Laboratories  
MATRIX: Sediments  
SAMPLE NUMBER: 97

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
MSL-M-42	40-120%	±20%	0.02µg/Kg

**HOLDING TIMES** Samples were held frozen up to 3 months prior to extraction and most were analyzed within the EPA extract holding time of 40 days (EPA 1986).

**DETECTION LIMITS** Detection limits ranged from 0.004 to 0.053 µg/g.

**METHOD BLANKS** Seven method blanks were extracted and analyzed for PAHs; one with each batch of samples. PAHs were not detected in the blanks.

**MATRIX SPIKES** Eight samples were spiked in duplicate with a known concentration of PAH standard. One sample had suspect results and was re-analyzed. However, the spikes were not re-analyzed with it, therefore, that data has not been included. A second sample had suspect results and was rerun along with the spikes. The rerun results were reasonable and have been included in the data package. For the 17 PAH compounds analyzed, spike 379GLBR\*111 had recoveries within the 40-120% criteria and the duplicate spike had 4 compounds within the criteria. The spike 379GLBR\*247 had 12 of 17 and the duplicate had 10 of 17 compounds within the criteria. Both the spike 379GLBR\*247 (rerun) and the duplicate had 14 of 17 compounds within the criteria. The spike 379GLBR\*268 had 8 of 17 and the duplicate had 16 of 17 compounds within the criteria. Both the spike 379GLBR\*388 and the duplicate had 12 of 17 compounds within the criteria. The spike 379GLBR\*388 (rerun) had 14 of 17 and the duplicate had 13 of 17 compounds within the criteria.

**SRMs** SRMs certified for PAHs in sediments are not available.

**REPLICATES** Five samples were extracted and analyzed in triplicate. Of those triplicates, one had 17 of 17 compounds within the ±20 criteria, two had 14 of 17 and two had 11 of 17 within the criteria. Those compounds with higher RSDs were associated with low concentrations near the detection limits.

**SURROGATES** Four surrogates were added to all samples. Of the 97 samples analyzed, 41 had one surrogate outside the recovery criteria of 40-120%, 8 had two surrogates outside the criteria and 2 had three surrogates outside the criteria.

## QA/QC SUMMARY

PROGRAM: Buffalo River Pilot Project  
PARAMETER: Polychlorinated biphenyls (PCBs)  
LABORATORY: Battelle Marine Science Laboratories  
MATRIX: Sediments  
SAMPLE NUMBER: 46

<u>Reference Method</u>	<u>Range of Recovery</u>	<u>Relative Precision</u>	<u>Detection Limit</u>
MSL-M-42	40-120%	±20%	0.02µg/Kg

**HOLDING TIMES** Samples were held frozen for up to 3 months prior to extraction and were most were analyzed within the EPA extract holding time of 40 days (EPA 1986).

**DETECTION LIMITS** Detection limits ranged from 0.025 to 0.060µg/Kg.

**METHOD BLANKS** One method blank was extracted and analyzed with each batch. No PCBs were detected in the blanks.

**MATRIX SPIKES** One sample was spiked with Aroclor 1254. Four samples were spiked in duplicate with Aroclor 1254. Three of the seven recoveries were within the criteria of 40-120%.

**SRMs** Only one SRM is certified for PCBs in sediments, which is HS-2 from the National Research Council of Canada. However, only Aroclor 1254 is certified for packed column chromatography. We used capillary column chromatography. Therefore, no certified SRMs are available for PCBs in sediment using the method we follow.

**REPLICATES** Two samples were extracted and analyzed in triplicate. Sample 379GLBR\*199 had an RSD for Aroclor 1254 of 89%, and an RPD of 117% for Aroclor 1248. The second replicate, 379GLBR\*323 had RSDs within the criteria for all compounds.

**SURROGATES** Two surrogates were added to each sample. Thirty-three of 46 samples had one surrogate outside the criteria of 40-120%. Thirty-two of those 33 samples were outside the criteria because the amount of surrogate octachloronaphthalene added was too low relative to the sample concentrations and was difficult to resolve.

**BUFFALO RIVER PILOT PROJECT (CF# 379)  
DOXINS/FURANS IN AIR SAMPLES**

(Concentrations in ng/sample)

MSL Code	Sponsor ID	2378-		TOTAL		2378-		TOTAL		12378-		TOTAL		23478-		TOTAL		12378-		TOTAL		123478-		TOTAL		123678-		123789-	
		TCDF	TCDF	TCDF	TCDF	TCDD	TCDD	TCDD	TCDD	PeCDF	PeCDF	PeCDF	PeCDF	PeCDF	PeCDF	PeCDF	PeCDF	PeCDD	PeCDD	PeCDD	PeCDD	HxCDF	HxCDF	HxCDF	HxCDF	HxCDF	HxCDF	HxCDF	HxCDF
379GLBR-112ABE	22OCT5:0011GA10	0.086 U	0.100	0.079 U	0.079 U	NA	0.033	0.056 U	0.380	0.330	0.330	0.320	0.053	0.042	0.042	0.042	0.330	0.330	0.320	0.320	0.053	0.053	0.046	0.046	0.076	0.076	0.150	0.150	
379GLBR-112F	22OCT5:0011GA10	0.057	0.260	0.027 U	0.027 U	NA U	0.031	0.048	0.400	0.023 U	0.023 U	0.150	0.046	0.043	0.043	0.043	0.150	0.150	0.150	0.150	0.046	0.046	0.076	0.076	0.150	0.150	0.220	0.220	
379GLBR-232ABD	23OCT4:4511GA20	0.037	0.480	0.036	0.036	0.290	0.040	0.056	0.530	0.079	0.079	0.670	0.076	0.078	0.078	0.078	0.670	0.670	0.670	0.670	0.076	0.076	0.150	0.150	0.220	0.220	0.403	0.403	
379GLBR-233ABD	24OCT5:3011GA30	0.000	0.830	0.041	0.041	0.400	0.057	0.087	1.200	0.086	0.086	0.770	0.130	0.140 U	0.140 U	0.140 U	0.490	0.490	0.490	0.490	0.054	0.054	0.330	0.330	0.230	0.230	0.0058 U	0.0058 U	
379GLBR-234ABD	25OCT1:4511GB10	0.059	1.100	0.021	0.021	0.210	0.055	0.063	0.840	0.036 U	0.036 U	0.490	0.054	0.048	0.048	0.048	0.490	0.490	0.490	0.490	0.054	0.054	0.330	0.330	0.230	0.230	0.0058 U	0.0058 U	
379GLBR-272ABD	31OCT1:0011GB30	0.380	3.800	0.089	0.089	1.100	0.360	0.340	3.700	0.180 U	0.180 U	2.600	0.330	0.390	0.390	0.390	0.180 U	0.180 U	2.600	2.600	0.330	0.330	0.230	0.230	0.0058 U	0.0058 U	0.0058 U	0.0058 U	
Method Blank	Method Blank	0.0260 U	NA	0.0530 U	0.0530 U	NA	0.0120 U	0.0082 U	NA	0.0110 U	0.0110 U	NA	0.0094 U	0.0076 U	0.0076 U	0.0076 U	NA	0.0110 U	NA	NA	0.0094 U	0.0094 U	0.0094 U	0.0094 U	0.0058 U	0.0058 U	0.0058 U	0.0058 U	

**MATRIX SPIKE RESULTS**

**Spike**

Quantity Spiked	0.200	0.200	0.200	0.200	0.200	0.200	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
Quantity Measured	0.230	0.230	0.250	0.250	0.250	0.250	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100
Percent Recovery	115%	115%	125%	125%	125%	125%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%

**Spike Duplicate**

Quantity Spiked	0.200	0.200	0.200	0.200	0.200	0.200	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
Quantity Measured	0.240	0.240	0.240	0.240	0.240	0.240	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100	1.100
Percent Recovery	120%	120%	120%	120%	120%	120%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%	110%

NA - Not applicable.

. - Outside of Internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (CF# 379)  
DOXINS/FURANS IN AIR SAMPLES**

(Concentrations in ng/sample)

MSL Code	Sponsor ID	234678-		123478-		123678-		123789-		1234678-		TOTAL	
		HxCDF	TOTAL	HxCDF	TOTAL	HxCDF	TOTAL	HxCDF	TOTAL	HxCDF	TOTAL	HxCDF	TOTAL
379GLBR-112ABE	22OCT5:0011GA10	0.076 U	0.560	0.050	0.091	0.050	0.130	0.510	0.100	0.710	1.600	3.300	5.200
379GLBR-112F	22OCT5:0011GA10	0.042	0.590	0.013 U	0.022	0.011 U	0.100	0.620	0.120	1.200	0.210	0.390	0.910
379GLBR-232ABD	23OCT4:4511GA20	0.028	0.890	0.031	0.050	0.025	0.660	0.750	0.110	1.100	0.450	0.900	1.600
379GLBR-233ABD	24OCT5:3011GA30	0.054	1.100	0.049	0.071	0.050	1.000	1.200	0.180	1.900	0.780	2.300	3.000
379GLBR-234ABD	25OCT1:4511GB10	0.023 U	0.440	0.026 U	0.038 U	0.020	0.490	0.250	0.023	0.410	0.260	0.680	1.300
379GLBR-272ABD	31OCT1:0011GB30	0.044	2.800	0.120	0.130	0.110	2.900	1.600	0.110	1.900	1.500	4.000	4.900
Method Blank	Method Blank	0.0077 U	NA	0.0083 U	0.0097 U	0.0120 U	NA	0.0190 U	0.0077 U	NA	NA	NA 0.0210	NA

## MATRIX SPIKE RESULTS

## Spike

	1.000	4.000	1.000	1.000	3.000	1.000	1.000	2.000	1.000	2.000	2.000
Quantity Spiked	1.000	4.000	1.000	1.000	3.000	1.000	1.000	2.000	1.000	2.000	2.000
Quantity Measured	0.980	3.700	1.200	1.300	3.700	1.100	1.600	2.700	0.970	2.300	2.100
Percent Recovery	98%	93%	120%	130%	123%	110%	160%	135%	97%	115%	105%

## Spike Duplicate

	1,000	4,000	1,000	1,000	1,000	3,000	1,000	1,000	2,000	1,000	1,000	2,000	2,000
Quantity Spiked	1,000	4,000	1,000	1,000	1,000	3,000	1,000	1,000	2,000	1,000	1,000	2,000	2,000
Quantity Measured	1,000	3,800	1,200	0,990	0,990	3,200	1,200	1,600	2,800	1,100	1,100	2,400	2,100
Percent Recovery	100%	95%	120%	99%	99%	107%	120%	160%	140%	110%	110%	120%	105%

**NA - Not applicable.**

• = Outside of Internal QC criteria (40-120%).



**BUFFALO RIVER PILOT PROJECT (CF# 379)  
DOXINS/FURANS IN AIR SAMPLES**

**INTERNAL STANDARDS**

% Recovery																									
MSL Code	Sponsor ID	2378-		2378-		23478-		12378-		12378-		123478-		123678-		234678-		123789-		123478-		123678-			
		TCDF -C13	TCDF -C13	TCDF -C13	TCDF -C13	PeCDF -C13	PeCDF -C13	PeCDF -C13	PeCDF -C13	PeCDF -C13	PeCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13	HxCDF -C13			
379GLBR-112ABE	22OCT5:0011GA10	92	93	70	85	66	106	83	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	122	123478- -C13	123678- -C13			
379GLBR-112F	22OCT5:0011GA10	78	73	63	NA	37	NA	86	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	80				
379GLBR-232ABD	23OCT4:4511GA20	93	88	73	111	42	93	93	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	115	83				
379GLBR-233ABD	24OCT5:3011GA30	104	101	79	103	62	93	102	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	145	89				
379GLBR-234ABD	25OCT1:4511GB10	78	77	61	113	40	86	79	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	115	73				
379GLBR-272ABD	31OCT1:0011GB30	88	88	68	100	37	89	88	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	148	73				
Method Blank	Method Blank	85	80	73	124	57	96	92	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	120	77				

**MATRIX SPIKE RESULTS**

Spike	(% Recovery)																								
Spike Duplicate	(% Recovery)	81	84	77	103	46	94	88	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
		75	71	60	109	36	90	82	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

NA = Not applicable.  
\* = Outside of Internal QC criteria (40-120%).

BUFFALO RIVER PILOT PROJECT (CF# 379)  
DOXINS/FURANS IN AIR SAMPLES

% Recovery		1234678-1234789-1234678-			1234-123789-			2378-	
MSL Code	Sponsor ID	HxCDF -C13	HxCDF -C13	HxCDF -C13	OCDD -C13	TCDD -C13	HxCDD -C13	TCDD -C13	TCDD -C13
379GLBR-112ABE	22OCT5:0011GA10	77	119	87	102	NA	NA	100	100
379GLBR-112F	22OCT5:0011GA10	69	NA	73	61	NA	NA	NA	NA
379GLBR-232ABD	23OCT4:4511GA20	74	108	83	83	NA	NA	99	99
379GLBR-233ABD	24OCT5:3011GA30	82	108	91	95	NA	NA	101	101
379GLBR-234ABD	25OCT1:4511GB10	64	110	73	59	NA	NA	99	99
379GLBR-272ABD	31OCT1:0011GB30	70	108	76	74	NA	NA	95	95
Method Blank	Method Blank	76	105	72	59	NA	NA	100	100
METHOD BLANK RESULTS									
Spike	(% Recovery)	53	135	77	67	NA	NA	93	93
Spike Duplicate	(% Recovery)	44	135	63	50	NA	NA	100	100

NA = Not applicable.  
\* = Outside of internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)  
SIR/PAH ANALYSIS  
IN AIR SAMPLES**

**MATRIX: XAD** (Concentrations in ng/sample)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Flourene	Phenanthrene	Anthracene	Fluoranthene	Pyrene
379GLBR-112ABE	22OCT5:0011GA10	155261 BD(1)	7529 D	14897 D(1)	6766 D	51605 D(1)	5492 D	12605 D	22876 D(1)
379GLBR-232ABD	23OCT4:4511GA20	19086 BD(2)	82	305	448	3568 D(2)	330	1289	728
379GLBR-233ABD	24OCT5:3011GA30	13944 BD(2)	168	414	470	4044 D(2)	477	1650	1296
379GLBR-234ABD	25OCT1:4511G310	8922 BD(2)	187	478	854	3743 D(2)	813	1779 D(2)	2691
379GLBR-272ABD	31OCT1:0011GB30	111623 BD(1)	541 D	1663 D	3841 D	44750 D(1)	4423 D	32187 D(1)	11945 D
Method Blank-XAD		77	60 U	60 U	60 U	60 U	60 U	60 U	60 U

**MATRIX SPIKE RESULTS**

Amount Spiked	1500	1500	1500	1500	1500	1500	1500	1500	1500
Method Blank	77	60 U	60 U	60 U	60 U	60 U	60 U	60 U	60 U
Blank + Spike	1137	1097	1395	1395	1154	1159	957	1381	1211
Amount Recovered	1060	1097	1395	1395	1154	1159	957	1381	1211
Percent Recovery	71%	73%	93%	93%	77%	77%	64%	92%	81%
Amount Spiked	1500	1500	1500	1500	1500	1500	1500	1500	1500
Method Blank	77	60 U	60 U	60 U	60 U	60 U	60 U	60 U	60 U
Blank + Spike Duplicate	1573	1401	1973	1973	1662	1615	1324	1962	1689
Amount Recovered	1496	1401	1973	1973	1662	1615	1324	1962	1689
Percent Recovery	100%	93%	132%*	132%*	111%	108%	88%	131%*	113%

# - All benzofluoranthene isomers (b, j & k) are quantified together  
B - Analyte was also detected in the associated blank  
D - Secondary analysis performed at dilution factor of 1:5  
D(1) - Third analysis performed at dilution factor of 1:100  
D(2) - Secondary analysis performed at dilution factor of 1:20  
U - Undetected at the given method detection limit  
\* - Outside EPA and Internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)**  
**SIR/PAH ANALYSIS**  
**IN AIR SAMPLES**

**MATRIX: XAD** (Concentrations in ng/sample)

MSL Code	Sponsor ID	Benz[a]- anthracene	Chrysene	Benzofluor- anthenes #	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene	Dibenz[a,h]- anthracene	Benzo[ghi] perylene
379GLBR-112ABE	22OCT5:0011GA10	54520 D	5370 D	2606 D	2956 D	711 D	666 D	3118 D
379GLBR-232ABD	23OCT4:4511GA20	128	159	313	69	60 U	60 U	77
379GLBR-233ABD	24OCT5:3011GA30	268	286	363	124	64	60 U	97
379GLBR-234ABD	25OCT1:4511GB10	946	699	839	223	214	60 U	121
379GLBR-272ABD	31OCT1:0011GB30	5011 D	3629 D	6656 D	805 D	1234 D	60 U	434 D
Method Blank-XAD		60 U	60 U	120 U	60 U	60 U	60 U	60 U

**MATRIX SPIKE RESULTS**

Amount Spiked	1500	1500	1500	1500	1500	1500	1500	1500
Method Blank	60 U	60 U	60 U	120 U	60 U	60 U	60 U	60 U
Blank + Spike	1225	1153	1153	2611	804	1292	1331	1215
Amount Recovered	1225	1153	1153	2611	804	1292	1331	1215
Percent Recovery	82%	77%	77%	87%	54%	86%	89%	81%
Amount Spiked	1500	1500	1500	1500	1500	1500	1500	1500
Method Blank	60 U	60 U	60 U	120 U	60 U	60 U	60 U	60 U
Blank + Spike Duplicate	1708	1579	1579	3749	1000	1812	1852	1678
Amount Recovered	1708	1579	1579	3749	1000	1812	1852	1678
Percent Recovery	114%	105%	105%	125% *	67%	121% *	123% *	112%

# = All benzofluoranthene isomers (b, j & k) are quantified together  
 B = Analyte was also detected in the associated blank  
 D = Secondary analysis performed at dilution factor of 1:5  
 D(1) = Third analysis performed at dilution factor of 1:100  
 D(2) = Secondary analysis performed at dilution factor of 1:20  
 U = Undetected at the given method detection limit  
 \* = Outside EPA and Internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)  
SIR/PAH ANALYSIS  
IN AIR SAMPLES**

**MATRIX: XAD**

MSL Code	Sponsor ID	% Surrogate Recovery		
		D10- Fluorene	D10- Anthracene	D10- Pyrene

379GLBR-112ABE	22OCT5:0011GA10	162% D *	107% D	204% D *
379GLBR-232ABD	23OCT4:4511GA20	60%	66%	65%
379GLBR-233ABD	24OCT5:3011GA30	58%	63%	67%
379GLBR-234ABD	25OCT1:4511GB10	108%	113%	116%
379GLBR-272ABD	31OCT1:0011GB30	94% D	101% D	111% D

Method Blank-XAD

103% 79% 109%

**MATRIX SPIKE RESULTS**

Amount Spiked	NA	NA	NA	NA
Method Blank	103%	79%	109%	109%
Blank + Spike	77%	63%	86%	86%
Amount Recovered	NA	NA	NA	NA
Percent Recovery	NA	NA	NA	NA

Amount Spiked	NA	NA	NA	NA
Method Blank	103%	79%	109%	109%
Blank + Spike Duplicate	107%	87%	120%	120%
Amount Recovered	NA	NA	NA	NA
Percent Recovery	NA	NA	NA	NA

# - All benzofluoranthene isomers (b, j & k) are quantified together

B - Analyte was also detected in the associated blank

D - Secondary analysis performed at dilution factor of 1:5

D(1) - Third analysis performed at dilution factor of 1:100

D(2) - Secondary analysis performed at dilution factor of 1:20

U - Undetected at the given method detection limit

\* - Outside EPA and internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)  
SIR/PAH ANALYSIS  
IN AIR SAMPLES**

**MATRIX: WATER** (Concentrations in ng/L)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Flourene	Phenanthrene	Anthracene	Fluoranthene	Pyrene
379GLBR-112CD	22OCT5:0011GA10	299	41	103	123	2975 D	233	1433 D	4602 D
379GLBR-232C	23OCT4:4511GA20	98	45 U	45 U	45 U	61	45 U	45 U	46
379GLBR-233C	24OCT5:3011GA30	56	50 U	50 U	50 U	55	50 U	50 U	55
379GLBR-234C	25OCT1:4511GB10	92	57 U	57 U	57 U	96	57 U	187	186
379GLBR-272C	31OCT1:0011GB30	93	74 U	74 U	74 U	74 U	74 U	74 U	74 U
Method Blank-WATER		20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U

**MATRIX: SOLVENT BLANK** (Concentrations in ng/L)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Flourene	Phenanthrene	Anthracene	Fluoranthene	Pyrene
379GLBR-112F	22OCT5:0011GA10	4571 B	400 U	633	1472	10686	400 U	2544	1132

# = All benzofluoranthene isomers (b, j & k) are quantified together

B = Analyte was also detected in the associated blank

D = Secondary analysis performed at dilution factor of 1:5

U = Undetected at the given method detection limit

I = Interference present

\* = Outside EPA and Internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)  
SIR/PAH ANALYSIS  
IN AIR SAMPLES**

**MATRIX: WATER** (Concentrations in ng/L)

MSL Code	Sponsor ID	Benz[a]-anthracene	Chrysene	Benzofluoranthenes #	Benz[a]-pyrene	Indeno [1,2,3-cd]-pyrene	Dibenz[a,h]-anthracene	Benzo[ghi]perylene
379GLBR-112CD	22OCT5:0011GA10	647	890	235	149	57	70	170
379GLBR-232C	23OCT4:4511GA20	45 U	45 U	91 U	45 U	45 U	45 U	45 U
379GLBR-233C	24OCT5:3011GA30	50 U	50 U	100 U	50 U	50 U	50 U	50 U
379GLBR-234C	25OCT1:4511GB10	57 U	57 U	114 U	57 U	57 U	57 U	57 U
379GLBR-272C	31OCT1:0011GB30	74 U	74 U	148 U	74 U	74 U	74 U	74 U
Method Blank-WATER		20 U	20 U	40 U	20 U	20 U	20 U	20 U

**MATRIX: SOLVENT BLANK** (Concentrations in ng/L)

MSL Code	Sponsor ID	Benz[a]-anthracene	Chrysene	Benzofluoranthenes #	Benz[a]-pyrene	Indeno [1,2,3-cd]-pyrene	Dibenz[a,h]-anthracene	Benzo[ghi]perylene
379GLBR-112F	22OCT5:0011GA10	400 U	400 U	800 U	400 U	400 U	400 U	400 U

# = All benzofluoranthene isomers (b, j & k) are quantified together

B = Analyte was also detected in the associated blank

D = Secondary analysis performed at dilution factor of 1:5

U = Undetected at the given method detection limit

I = Interference present

\* = Outside EPA and Internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)  
SIR/PAH ANALYSIS  
IN AIR SAMPLES**

**MATRIX: WATER**

MSL Code	Sponsor ID	% Surrogate Recovery		
		D10-Fluorene	D10-Anthracene	D10-Pyrene
379GLBR-112CD	22OCT5:0011GA10	89%	85%	149% <sup>*</sup>
379GLBR-232C	23OCT4:4511GA20	74%	79%	89%
379GLBR-233C	24OCT5:3011GA30	52%	54%	68%
379GLBR-234C	25OCT1:4511GB10	75%	71%	87%
379GLBR-272C	31OCT1:0011GB30	69%	65%	87%
Method Blank-WATER		52%	35% <sup>*</sup>	72%

**MATRIX: SOLVENT BLANK**

MSL Code	Sponsor ID	% Surrogate Recovery		
		D10-Fluorene	D10-Anthracene	D10-Pyrene
379GLBR-112F	22OCT5:0011GA10	101%	80%	116%

# = All benzo(a)fluoranthene isomers (b, j & k) are quantified together

B = Analyte was also detected in the associated blank

D = Secondary analysis performed at dilution factor of 1:5

U = Undetected at the given method detection limit

i = Interference present

\* = Outside EPA and internal QC criteria (40-120%).



**BUFFALO RIVER PILOT PROJECT (#379)  
PCB/PESTICIDE ANALYSIS  
IN AIR SAMPLES**

MATRIX: XAD		(Concentrations in ug/sample)							
MSL Code	Sponsor ID	Monochloro- biphenyl	Dichloro- biphenyl	Trichloro- biphenyl	Tetrachloro- biphenyl	Pentachloro- biphenyl	Hexachloro- biphenyl	Heptachloro- biphenyl	Octachloro- biphenyl
379GLBR-112ABE	22OCT5:0011GA10	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U
379GLBR-232ABD	23OCT4:4511GA20	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U
379GLBR-233ABD	24OCT5:3011GA30	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U
379GLBR-234ABD	25OCT1:4511GB10	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U
379GLBR-272ABD	31OCT1:0011GB30	0.75 U	0.75 U	3.6	2.3	2.9	1.5 U	2.3 U	2.3 U
Method Blank-XAD		0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U

**MATRIX SPIKE RESULTS**

Amount Spiked	12	12	12	12	24	24	24	36	36
Method Blank	0.75 U	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U
Blank + Spike	6.6	7.8	8.7	8.7	18	17	18	27	27
Amount Recovered	6.6	7.8	8.7	8.7	18	17	18	27	27
Percent Recovery	55%	65%	73%	73%	75%	71%	75%	75%	75%
Amount Spiked	12	12	12	12	24	24	24	36	36
Method Blank	0.75 U	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	2.3 U	2.3 U
Blank + Spike Duplicate	7.3	7.7	7.8	7.8	16	13	13	19	19
Amount Recovered	7.3	7.7	7.8	7.8	16	13	13	19	19
Percent Recovery	61%	64%	65%	65%	67%	54%	54%	53%	53%

MATRIX: SOLVENT BLANK		(Concentrations in ug/L)							
MSL Code	Sponsor ID	Monochloro- biphenyl	Dichloro- biphenyl	Trichloro- biphenyl	Tetrachloro- biphenyl	Pentachloro- biphenyl	Hexachloro- biphenyl	Heptachloro- biphenyl	Octachloro- biphenyl
379GLBR-112F	22OCT5:0011GA10	5.0 U	5.0 U	5.0 U	10 U	10 U	10 U	15 U	15 U

U = Undetected at the given method detection limit.  
• = Outside EPA and Internal QC criteria (40-120%).

**BUFFALO RIVER PILOT PROJECT (#379)  
PCB/PESTICIDE ANALYSIS  
IN AIR SAMPLES**

MATRIX: XAD		(Concentrations in ug/sample)		% Surrogate Recovery	
MSL Code	Sponsor ID	Nonachloro- biphenyl	Decachloro- biphenyl	2-Fluoro- biphenyl	C13-Tetrachloro- biphenyl C13-Octachloro- biphenyl
379GLBR-112ABE	22OCT5:0011GA10	2.3 U	3.8 U	62%	71% 59%
379GLBR-232ABD	23OCT4:4511GA20	2.3 U	3.8 U	81%	107% 96%
379GLBR-233ABD	24OCT5:3011GA30	2.3 U	3.8 U	77%	112% 105%
379GLBR-234ABD	25OCT1:4511GB10	2.3 U	3.8 U	60%	94% 92%
379GLBR-272ABD	31OCT1:0011GB30	2.3 U	3.8 U	53%	95% 88%
Method Blank-XAD		2.3 U	3.8 U	23% *	67% 64%

**MATRIX SPIKE RESULTS**

Amount Spiked	NS	60	NA	NA	NA
Method Blank	NS	3.8 U	23% *	67%	64%
Blank + Spike	NS	45	55%	97%	92%
Amount Recovered	NS	45	NA	NA	NA
Percent Recovery	NS	75%	NA	NA	NA
Amount Spiked	NS	60	NA	NA	NA
Method Blank	NS	3.8 U	23% *	67%	64%
Blank + Spike Duplicate	NS	32	64%	71%	67%
Amount Recovered	NS	32	NA	NA	NA
Percent Recovery	NS	53%	NA	NA	NA

MATRIX: SOLVENT BLANK		(Concentrations in ug/L)		% Surrogate Recovery	
MSL Code	Sponsor ID	Nonachloro- biphenyl	Decachloro- biphenyl	2-Fluoro- biphenyl	C13-Tetrachloro- biphenyl C13-Octachloro- biphenyl
379GLBR-112F	22OCT5:0011GA10	15	25	47%	93% 94%

U = Undetected at the given method detection limit.  
\* = Outside EPA and Internal QC criteria (40-120%).



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**REPORT OF: CHEMICAL ANALYSES**

**PROJECT:** PCB/PAH ANALYSES

**DATE:** January 9, 1992

**ISSUED TO:** Battelle Pacific Northwest Division  
Attn: Ms. Linda Bingler  
Marine Sciences Laboratory  
439 West Sequim Bay Road  
Sequim, WA 98382

**INVOICE NO:** 4410 92-0442B

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**INTRODUCTION**

This report summarizes the results from analyses performed on eleven samples which were submitted by a representative of Battelle Pacific Northwest Division. Six of the samples were analyzed for the presence or absence of polychlorinated biphenyls (PCBs) using a modified version of USEPA Method 680 and all of the samples were analyzed for polynuclear aromatic hydrocarbons (PAHs) using a version of USEPA Method 8270 adapted for selected-ion-monitoring analyses.

**SAMPLE IDENTIFICATION**

<u>Client ID</u>	<u>Sample Type</u>	<u>Analyses</u>	<u>TCT ID</u>
379GLBR*112ABE	Air	PCB/PAH	268734
379GLBR*112CD	Impinger	PAH	268743
379GLBR*112F	Blank Solvent	PCB/PAH	268741
379GLBR*232ABD	Air	PCB/PAH	268735
379GLBR*232C	Impinger	PAH	268744
379GLBR*233ABD	Air	PCB/PAH	268736
379GLBR*233C	Impinger	PAH	268750
379GLBR*234ABD	Air	PCB/PAH	268737
379GLBR*234C	Impinger	PAH	268751
379GLBR*272ABD	Air	PCB/PAH	268738
379GLBR*272C	Impinger	PAH	268752

## REPORT OF: CHEMICAL ANALYSES

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### METHODOLOGY

#### Sample Extraction

The XAD-2 resin and filter from each air sample component set were combined in a Soxhlet extractor and spiked with the following surrogate standard compounds:

	<u>µg Spiked</u>
2-Fluorobiphenyl	30
<sup>14</sup> C <sub>12</sub> -Tetrachlorobiphenyl	9.6
<sup>14</sup> C <sub>12</sub> -Octachlorobiphenyl	9.6
<sup>2</sup> H <sub>10</sub> -Fluorene	1.5
<sup>2</sup> H <sub>10</sub> -Anthracene	1.5
<sup>2</sup> H <sub>10</sub> -Pyrene	1.5

Each sample was extracted with methylene chloride and each extract was combined with the respective train rinse solvent component. The extracts were transferred to Kuderna-Danish flasks, concentrated to 3.0 mL, and split into three equal portions for PCB, PAH, and PCDD/PCDF analyses. (The results from the PCDD/PCDF analyses were reported previously in TCT report #4410 92-0442A.) The PCB portions were solvent exchanged to hexane and spiked with 10 µg of each of the following perdeuterated internal standards:

<sup>1</sup>H<sub>12</sub>-Phenanthrene  
<sup>1</sup>H<sub>12</sub>-Chrysene

The impinger catch samples were spiked with 0.5 µg of each of the three perdeuterated PAH surrogates listed above, extracted with methylene chloride in separatory funnels, and the extracts were concentrated to 1.0 mL using Kuderna-Danish glassware. All of the final PAH extracts were then spiked with 0.5 µg of each of the following perdeuterated internal standards:

<sup>2</sup>H<sub>8</sub>-Naphthalene  
<sup>2</sup>H<sub>10</sub>-Acenaphthene  
<sup>2</sup>H<sub>10</sub>-Phenanthrene  
<sup>2</sup>H<sub>12</sub>-Chrysene  
<sup>2</sup>H<sub>12</sub>-Perylene

## REPORT OF: CHEMICAL ANALYSES

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### PCB Analyses

PCB analyses were performed according to a modified version of USEPA Method 680. A 1-uL aliquot of each sample extract was injected by autosampler onto a 30 m DB-5 capillary column in a Hewlett-Packard Model 5890A gas chromatograph. The column exit was interfaced directly into the ion source of a VG Trio-2 quadrupole mass spectrometer operating in the positive ion electron impact (EI) ionization mode at 70 eV. The GC/MS operating conditions for these analyses are listed in Table 1. Data were acquired in the selected-ion-monitoring mode and processed using a VG 11-250J data system.

A five point initial calibration curve was generated by analyzing standard solutions containing the target compounds at concentrations ranging from 0.5-125 ug/mL as shown in Table 2. Each solution contained internal standards at fixed concentrations of 10 ug/mL. Response factors were generated for each target analyte relative to the corresponding internal standard using the measured area responses for characteristic ions and the known concentrations. Continuing calibration check standards were analyzed daily prior to sample analysis in order to verify the validity of the initial calibration. All calculations were performed as specified in Method 680. The specific ions that were monitored for quantitation and confirmation of the PCB compounds are listed in Table 3.

### PAH Analyses

The PAH analyses were performed by selected-ion-monitoring (SIM) gas chromatography/mass spectrometry (GC/MS) using procedures from USEPA Method 8270. 2-uL aliquots of the sample extracts were injected by autosampler onto a 30 m DB-5 capillary column in a Hewlett-Packard Model 5890A gas chromatograph. The column exit was interfaced directly into the ion source of a VG Trio-2 quadrupole mass spectrometer operating in the positive ion electron impact (EI) ionization mode at 70 eV. Data were acquired in the selected-ion-monitoring mode and processed using a VG 11-250J data system. The GC/MS operating conditions for these analyses are summarized in Table 1A.

A five point initial calibration curve was generated by analyzing standard solutions containing each of the target analytes at levels ranging from 20-1000 ng/mL and the internal standards at fixed concentrations of 500 ng/mL as indicated in Table 2A. Quantifications of the target compounds were performed by comparing the integrated areas of the chromatographic peaks with those of the internal standards as specified in Method 8270. Continuing calibration standard analyses were performed daily prior to sample analyses in order to verify the validity of the initial calibration. The specific ions that were monitored for the PAH analyses are listed in Table 3A.

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DATE: January 13, 1992

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### RESULTS

The results from the analyses are included in the following appendices:

Appendix A	Chain of Custody Documentation
Appendix B	PCB Sample Analysis Results
Appendix C	PCB QA/QC Analysis Results
Appendix D	PCB Sample Chromatograms
Appendix E	PCB QA/QC Chromatograms
Appendix F	PAH Sample Analysis Results
Appendix G	PAH QA/QC Analysis Results
Appendix H	PAH Sample Chromatograms and Raw Data
Appendix I	PAH QA/QC Chromatograms and Raw Data

### DISCUSSION

The recoveries of the surrogate compounds in the PCB samples fell within the 50-125% guidelines specified in Method 680, with the exception of the 2-Fluorobiphenyl recovery (47%) in sample 379GLBR\*112F. The designated range for the surrogate compounds in the PAH samples is 50-150% for at least two of the three surrogates in each sample. All of the surrogate recoveries in the PAH samples fell within this range, with the exception of the perdeuterated pyrene (227%) in sample 379GLBR\*112ABE. This elevated recovery resulted from the presence of an interference in the sample extract that was not resolved from the surrogate peak. However, since the remaining two surrogates in this sample were recovered at acceptable levels, the recovery criteria were met and no corrective action was required. The only other deviation from the target QC ranges was that of the raw area count for the perdeuterated naphthalene internal standard in sample 379GLBR\*272ABD, which was 269% of the value for the corresponding standard in the daily continuing calibration analysis. (The target range for internal standard areas in the sample extracts is 50-200% of the daily continuing calibration values.) However, upon analysis of a dilution of this sample extract, the naphthalene internal standard area fell into the acceptable range.

A laboratory method blank was prepared and analyzed with each sample extraction batch as part of our routine quality control/quality assurance procedures. The results, found at the beginnings of Appendices C and G, show the blanks to be free of the target PCB and PAH analytes, with the exception of a trace background level (77 nanograms) of naphthalene in the blank associated with the PAH portions of the air

## REPORT OF: CHEMICAL ANALYSES

PROJECT: PCB/PAH ANALYSES

DATE: January 9, 1992

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### DISCUSSION (Continued)

samples. The naphthalene levels determined for the affected samples were all higher than the naphthalene level in the blank by 20 times or more. This indicates that the sample processing steps did not contribute significantly to the levels reported for the samples. A low recovery was achieved for the 2-Fluorobiphenyl surrogate in the PCB blank due to inadvertent volatilization of this compound during the concentration steps. Since the entire sample was consumed in the extraction process, any reprocessing in order to improve the recovery was not possible. Also, the blank associated with the impinger samples exhibited a low recovery (35%) for the perdeuterated anthracene surrogate. Since good recoveries were achieved for the other two surrogates in this blank, no corrective action was required.

Two laboratory quality control PCB/PAH spike samples were prepared with the air sample batch by extracting aliquots of clean resin that had been fortified with native standard materials. The results, included in Appendices C and G, show that the spiked native compounds were recovered at levels ranging from 52-132%, which are all within the 50-135% target ranges designated for these analyses.

### REMARKS

The sample extracts will be retained for a period of 60 days from the date of this report and then discarded unless other arrangements are made. The raw mass spectral data will be archived on magnetic tape for a period of not less than one year. Questions regarding the data contained in this report may be addressed to the authors at the numbers provided below.

### TWIN CITY TESTING CORPORATION

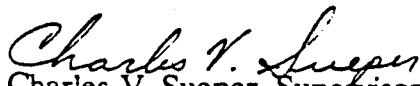


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**REPORT OF: CHEMICAL ANALYSES**

**PROJECT:** PCDD/PCDF ANALYSES

**DATE:** January 6, 1992

**ISSUED TO:** Battelle Pacific Northwest Division  
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Marine Sciences Laboratory  
Sequim, WA 98382

**INVOICE NO:** 4410 92-0442A

**INTRODUCTION**

This report summarizes the results from the analyses performed on six air samples which were submitted by a representative of Battelle Pacific Northwest Division. The samples were analyzed for the presence or absence of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) using a modified version of EPA Method 23. Polychlorinated biphenyl (PCB) and polynuclear aromatic hydrocarbons (PAH) data will be reported under separate cover.

**SAMPLE IDENTIFICATION**

<u>Client ID **</u>	<u>Sample Type</u>	<u>TCT ID</u>
379GLBR*112ABE	Air	268734
379GLBR*112F	Blank	268741
379GLBR*232ABD	Air	268735
379GLBR*233ABD	Air	268736
379GLBR*234ABD	Air	268737
379GLBR*272ABD	Air	268738

\*\* Samples and sample components were split as per instructions from Battelle personnel included in Appendix A.

**METHODOLOGY**

**PCDD/PCDF Extraction**

The XAD-2 resin portion of each sample component set was spiked with isotopically-labeled PCDD/PCDF internal standards (Table 1), combined with the filter, and placed in a Soxhlet extractor thimble. The train rinse solvents containing particulate were filtered and the filters were added to the respective Soxhlet extractor thimbles. The filtrate was then concentrated in the Soxhlet flask and the Soxhlet charged with methylene chloride. The sample components were extracted for eighteen hours and the methylene chloride was removed and concentrated. The Soxhlet was then recharged with benzene and extracted for an additional eighteen hours. One third of each of the methylene chloride and benzene extracts were then



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### PCDD/PCDF Extraction (Continued)

combined and quantitatively transferred to Kuderna Danish concentrators, concentrated, and solvent exchanged to hexane. The hexane extracts were then processed through the analyte enrichment procedures described below.

### Analyte Enrichment for PCDD/PCDF Analyses

The extraction procedure often removes a variety of compounds, in addition to the PCDDs and PCDFs, from the sample matrix. Some of these compounds can directly interfere with the analyses while others can overload the capillary column causing degradation in chromatographic resolution or sensitivity. The analyte enrichment steps described below were used to remove interferences from the extracts.

The extracts were diluted to 100 mL with hexane, transferred to separatory funnels, and washed with 1N sodium hydroxide, concentrated sulfuric acid, and distilled water. The hexane layers were concentrated to 1 mL and quantitatively transferred to liquid chromatography columns containing alternating layers of silica gel, 44% concentrated sulfuric acid on silica gel, and 33% 1 N sodium hydroxide on silica gel. The columns were eluted with 60 mL of hexane and each entire eluate was collected and concentrated, under a gentle stream of dry nitrogen, to a volume of 1 mL.

The extracts were then fractionated on liquid chromatography columns containing 4 g of activated alumina. The columns were eluted with 10 mL of hexane followed by 7 mL of 2.0% methylene chloride/hexane and 25 mL of 60% methylene chloride in hexane. The 60% methylene chloride/hexane fractions were concentrated to 1 mL under a stream of dry nitrogen and applied to the tops of chromatography columns containing 1 g of 5% AX-21 activated carbon on silica gel. Each column was eluted with cyclohexane/methylene chloride (50:50 V/V) and cyclohexane/methanol/benzene (75:20:5 V/V) in the forward direction, and then with benzene in the reverse direction. Each benzene fraction was collected, spiked with recovery standards (1,2,3,4-TCDD- $^{13}\text{C}_{12}$  and 1,2,3,7,8,9-HxCDD- $^{13}\text{C}_{12}$ ) and concentrated to a final volume of 20  $\mu\text{L}$ .

### PCDD/PCDF Analyses

The extracts were analyzed for the presence of PCDDs and PCDFs using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The instrumentation consisted of a Hewlett Packard Model 5890 gas chromatograph and a VG Model 70SE high resolution mass spectrometer. The capillary column was interfaced directly into the ion source of the mass spectrometer, thus providing the highest possible sensitivity while minimizing degradation of the chromatographic resolution.

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DATE: January 6, 1992

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### PCDD/PCDF Analyses (Continued)

The mass spectrometer was operated in the electron impact ionization mode at a mass resolution of 10,000-11,000 ( $M/\Delta M$ , 10 percent valley definition). This resolution is sufficient to resolve most interferences, such as PCBs, thus providing the highest level of confidence that the detected levels of PCDD/PCDF are not false positives resulting from interferences. Typical operating parameters for the HRGC/HRMS analyses are summarized in Table 2.

The data were acquired by selected-ion-recording (SIR) monitoring the groups of ion masses described in EPA method 23. The five groups corresponded to the tetrachlorinated through octachlorinated congener classes. Each group contained three ion masses for the PCDDs (with the exception of TCDD which contained two ion masses), two ion masses for the PCDFs, the corresponding ion masses from the two isotopically labeled internal standards, and the ion mass characteristic of the polychlorinated diphenylether (PCDPE) which, if present, could cause false responses in the dibenzofuran channels. The third PCDD ion mass monitored in the pentachloro through octachlorodibenzo-p-dioxin groups prevented the possibility of misinterpretation of a polychlorinated biphenylene isomer as a PCDD. The two ion masses monitored for TCDD also fulfilled this purpose.

Each group of ion masses also contained a lock mass which was monitored during the analyses to detect suppressive interferences. It is particularly important to detect this type of interference since it can cause the quantification of congener class levels to be artificially high if it occurs during the elution of an internal standard or low if it occurs during the elution of the native analytes.

The lock mass was also used by the data system to automatically correct the mass focus of the instrument. The data system determined the centroid of the lock mass during each data acquisition cycle and corrected the mass focus of the analyte and internal standard ion masses to assure that the centers of the mass peaks were being monitored.

The criteria used to judge positive responses for the PCDD/PCDF isomer included:

- Simultaneous response at both ion masses of the PCDD or PCDF
- Signal to noise ratio equal to or greater than 2.5:1.0 for both ion masses
- Chlorine isotope ratio within 15% of the theoretical value

## REPORT OF: CHEMICAL ANALYSES

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### PCDD/PCDF Analyses (Cont.)

- Chromatographic retention time within -1 to +3 seconds of the authentic standards (where applicable)
- Chromatographic retention times within elution windows determined from analyses of standard mixtures
- Absence of simultaneous response between the PCDF and diphenylether ion traces

A list of the exact ion masses monitored for the determination of PCDD/PCDF isomers and the PCDF interferences is presented in Table 3. Also included are the theoretical chlorine isotope ratios for the ten congener classes.

### Quantification and Calculations

The PCDD/PCDF isomers were quantified by comparison of their responses to the responses of the labeled internal standards as described EPA Method 23. Relative response factors were calculated from analyses of standard mixtures containing representatives of each of the PCDD/PCDF congener classes at five concentration levels, and each of the internal standards at one concentration level, as shown in Table 4. The PCDD/PCDF response factors were calculated by comparing the sum of the responses from the two ion masses monitored for each chlorine congener class to the sum of the responses from the two ion masses of the corresponding isotopically labeled internal standard. Table 5 shows the response factor at each of the calibration levels as well as the average response factors and the relative percent deviation for each. The formula for the response factor calculation is:

$$Rf = \frac{A_n \times Q_{is}}{A_{is} \times Q_n}$$

where:

Rf = Response factor  
A<sub>n</sub> = Sum of integrated areas for native isomer  
Q<sub>is</sub> = Quantity of labeled internal standard  
A<sub>is</sub> = Sum of integrated areas for labeled internal standard  
Q<sub>n</sub> = Quantity of native isomer

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### Quantification and Calculations (Cont.)

The levels of PCDD/PCDF in the samples were quantified using the following equation:

$$C = \frac{A_n \times Q_{is}}{A_{is} \times W \times R_f}$$

where:

C = Concentration of target isomer or congener class  
A<sub>n</sub> = Sum of integrated areas for the target isomer or congener class  
Q<sub>is</sub> = Quantity of labeled internal standard added to the sample  
A<sub>is</sub> = Sum of integrated areas for the labeled internal standard  
W = Sample weight, volume or area  
R<sub>f</sub> = Response factor

Each pair of ion mass peaks in the selected-ion-current chromatograms was evaluated manually to determine if it met the criteria for a PCDD or PCDF isomer. Areas of all peaks exhibiting correct ion ratios and having retention times within the correct windows were then summed for calculations of total congener concentrations. A summary of the high resolution initial calibration chlorine isotope ratios is presented in Table 6. The toxic equivalency factors used to calculate the 2,3,7,8-TCDD equivalency are listed in Table 7.

A limit of detection (LOD) based on producing a signal that is 2.5 times the noise level, was calculated for each undetected 2,3,7,8-substituted isomer of any tetra through octa chlorinated congener class. The noise heights used to calculate the detection limits were measured at the retention time of the specific isomer. The formula used for calculating the LOD is:

$$LOD = \frac{H_n \times Q_{is} \times 2.5}{H_{is} \times W \times R_f}$$

where:

LOD=Single isomer limit of detection  
H<sub>n</sub> =Sum of noise heights at native isomer retention time  
Q<sub>is</sub> =Quantity of labeled internal standard added to the sample  
H<sub>is</sub> =Sum of peak heights for labeled internal standard  
W =Sample weight, volume or surface area  
R<sub>f</sub> =Response factor

## REPORT OF: CHEMICAL ANALYSES

PROJECT: PCDD/PCDF ANALYSES

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### Quantification and Calculations (Cont.)

The recovery of the 2,3,7,8-TCDD-<sup>37</sup>Cl<sub>4</sub> enrichment efficiency standard and each <sup>13</sup>C<sub>12</sub>-labeled internal standard, relative to either 1,2,3,4-TCDD-<sup>13</sup>C<sub>12</sub> or 1,2,3,7,8,9-HxCDD-<sup>13</sup>C<sub>12</sub> was calculated using the following equation:

$$\%R = \frac{A_{is} \times Q_{rs} \times 100\%}{R_{fr} \times A_{rs} \times Q_{is}}$$

where:

%R = Percent recovery of labeled internal standard

A<sub>is</sub> = Sum of integrated areas of labeled internal standards

Q<sub>rs</sub> = Quantity of recovery standard

A<sub>rs</sub> = Sum of integrated areas of recovery standard

R<sub>fr</sub> = Response factor of the specific labeled internal standard relative to the recovery standard

Q<sub>is</sub> = Quantity of the labeled internal standard congener added to the sample

### RESULTS

Sample analysis results are included in the following:

Appendix A - Chain of Custody Documentation

Appendix B - Sample Analysis Results

Appendix C - QA/QC and Daily Calibration Results

Appendix D - Sample Chromatograms and Raw Data

Appendix E - Standard Chromatograms and Raw Data

Appendix F - QA/QC Chromatograms and Raw Data

### DISCUSSION

The recoveries of the isotopically-labeled PCDD/PCDF internal and surrogate standards in the air samples generally ranged from 70-130%, indicating a high level of efficiency through the extraction and enrichment steps. Somewhat lower recovery values were obtained for selected internal standards in each of the samples, many due to the presence of interfering substances in the final sample extracts. These interferences, evidenced by suppressions in the lock mass ion traces, caused short-term fluctuations in the

## REPORT OF: CHEMICAL ANALYSES

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### DISCUSSION (Continued)

sensitivity of the mass spectrometer, thereby decreasing the apparent recoveries for the affected standards. (Actual recoveries are typically consistent throughout a given sample or show gradual trends instead of abrupt changes.) All of the recoveries were, however, within the target ranges specified in the method, with the exceptions of the labeled 1,2,3,7,8-PeCDD in samples 379GLBR\*112F (37%), 379GLBR\*272ABD (37%), and the lab spike duplicate (36%). Also, slightly elevated recoveries were obtained for selected surrogate compounds in samples 379GLBR\*233ABD (labeled 1,2,3,4,7,8-HxCDD, 145%), 379GLBR\*272ABD (labeled 1,2,3,4,7,8-HxCDD, 148%), the laboratory spike (labeled 1,2,3,4,7,8,9-HpCDF, 135%), and the laboratory spike duplicate (labeled 1,2,3,4,7,8,9-HpCDF, 135%). The native 1,2,3,7,8-PeCDD concentrations should be accurate for these samples since quantitation is based on isotope dilution. The native 1,2,3,4,7,8-HxCDD and 1,2,3,4,7,8,9-HpCDF concentration values in these four samples may, however, be slightly elevated since both the native and surrogate isomers are compared to a separate labeled isomer.

A laboratory method blank was prepared and analyzed with the sample extraction batch as part of our routine quality control/quality assurance procedures. The data, included at the beginning of Appendix C, show the blank to be free of PCDDs and PCDFs, with the exceptions of trace background levels of PeCDD (19 picograms), HpCDD (6.3 picograms), and OCDD (92 picograms). The levels determined for the affected isomers in the actual samples were higher than the corresponding blank levels by 3-100 times. It should be noted, however, that levels less than five times higher than the background are not generally considered to be statistically different from the background.

Two quality control PCDD/PCDF spike samples were also prepared with the sample batch by extracting clean resin that had been fortified with native standard materials. The data, included in Appendix C, show that the spiked native compounds were recovered at levels ranging typically from 87-130%. The only compound with recoveries outside of this range was 1,2,3,4,7,8,9-HpCDF, which exhibited a recovery of 160% in each of the spike samples. It should be noted, however, that quality control ranges for native spike sample recoveries are not specified in the method.

**REPORT OF: CHEMICAL ANALYSES**

**PROJECT:**

**PCDD/PCDF ANALYSES**

**DATE:** January 6, 1992

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**INVOICE NO:** 4410 92-0442A

**REMARKS**

The sample extracts will be retained for a period of 60 days from the date of this report and then discarded unless other arrangements are made. The raw mass spectral data will be archived on magnetic tape for a period of not less than one year. Questions regarding the data contained in this report may be addressed to the authors at the numbers provided below.

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SWH/CVS/SDM/lml

**BUFFALO RIVER PILOT PROJECT (CF#379)  
OIL AND GREASE IN WATER SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE (mg/L)
<b>BIN A</b>		
379GLBR- 128 Rep 1	23OCT4:459LA2O	284.0
379GLBR- 128 Rep 2	23OCT4:459LA2O	287.0
379GLBR- 128 Rep 3	23OCT4:459LA2O	271.0
379GLBR- 132	23OCT5:2010LA2O	109.5
379GLBR- 163	25OCT10:2010LA3M	214.0
379GLBR- 165	25OCT10:009LA3C	112.0
379GLBR- 204	30OCT4:196LA3O	1.1 U
<b>BIN B</b>		
379GLBR- 187	25OCT3:0710LB1O	28.5
379GLBR- 226	31OCT10:129LB2O	62.0
379GLBR- 227	31OCT10:2110LB2O	69.5
379GLBR- 254	31OCT2:5710LB3M	25.0
379GLBR- 255	31OCT3:069LB3M	44.0
<b>BIN C</b>		
379GLBR- 286 Rep 1	18NOV5:559LC1O	72.4
379GLBR- 286 Rep 2	18NOV5:559LC1O	95.2
379GLBR- 286 Rep 3	18NOV5:559LC1O	102.4
379GLBR- 326	19NOV5:509LC2O	81.5
379GLBR- 331	19NOV5:0510LC2O	30.4
379GLBR- 361	20NOV2:193LC3O	2.0
379GLBR- 366	20NOV2:419LC3O	112.8
379GLBR- 370	20NOV3:0110LC3O	16.3
<b>BIN D</b>		
379GLBR- 398	21NOV3:2610LD1O	71.2
379GLBR 404	21NOV4:169LD1O	57.0
<b>DILUTION WATER</b>		
379GLBR- 275	6NOV12:003LO	1.1 U

U = Analyte detected below detection limits.



**BUFFALO RIVER PILOT PROJECT (CF#379)  
OIL AND GREASE IN WATER SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE (mg/L)
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**BLANKS**

BLANK-1	0.12
BLANK-2	0
BLANK-3	0
BLANK-4	0.67
BLANK-5	0
BLANK-6	0
BLANK-7	0.80
BLANK-8	0

**MATRIX SPIKE RESULTS**

**% RECOVERY**

379GLBR- 128 *	23OCT4:459LA2O	280.7	
379GLBR- 128 MATRIX SPIKE		347.5	134% * *
379GLBR- 128 MATRIX SPIKE DUPLICATE		335.0	109%
379GLBR- 132	23OCT5:2010LA2O	109.5	
379GLBR- 132 MATRIX SPIKE		157.5	96%
379GLBR- 132 MATRIX SPIKE DUPLICATE		157.0	95%

**REPLICATE ANALYSES**

379GLBR- 128 Rep 1	23OCT4:459LA2O	284.0	
379GLBR- 128 Rep 2	23OCT4:459LA2O	287.0	
379GLBR- 128 Rep 3	23OCT4:459LA2O	271.0	
	RSD %	3%	
379GLBR- 286 Rep 1	18NOV5:559LC10	72.4	
379GLBR- 286 Rep 2	18NOV5:559LC10	95.2	
379GLBR- 286 Rep 3	18NOV5:559LC10	102.4	
	RSD %	17%	

U = Analyte detected below detection limits.

\* = Mean of replicated sample.

\*\* = Outside of QC criteria (40-120%).

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF#379)**  
**OIL AND GREASE IN SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE ug/g
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**BIN A**

379GLBR- 13	07OCT12:341SAO	1211.6
379GLBR- 16	07OCT12:401SAO	1389.2
379GLBR- 17	07OCT12:091SAO	1644.3
379GLBR- 57	09OCT11:502SA3O	1765.7
379GLBR- 58	09OCT11:382SA2O	1819.5
379GLBR- 59	09OCT11:282SA1O	1426.2
379GLBR- 104	22OCT11:317SA1O	7232.5
379GLBR- 107	22OCT11:378SA1O	3133.0
379GLBR- 108	22OCT4:203SA2O	2565.5
379GLBR- 111 Rep 1	22OCT5:563SA2O	2867.7
379GLBR- 111 Rep 2	22OCT5:563SA2O	1839.4
379GLBR- 111 Rep 3	22OCT5:563SA2O	2283.9
379GLBR- 113	23OCT1:303SA2O	1855.0
379GLBR- 122	23OCT4:284SA2O	94.6 U R
379GLBR- 123	23OCT4:274SA2O	306.2 U R
379GLBR- 124	23OCT4:294SA2O	300.9 U R
379GLBR- 136	24OCT10:457SA2O	2390.8
379GLBR- 139	24OCT10:508SA2O	696.4
379GLBR- 141	24OCT2:203SA3O	1690.4
379GLBR- 143	24OCT5:503SA3O	2669.4
379GLBR- 145	24OCT6:203SA3O	2390.8
379GLBR- 151	25OCT9:224SA3O	632.8 R
379GLBR- 152	25OCT9:224SA3O	490.2 U
379GLBR- 153	25OCT9:214SA3O	537.7 U
379GLBR- 166	25OCT9:307SA3O	2442.7
379GLBR- 167	25OCT9:368SA3O	1900.9

U = Analyte detected below detection limits.  
R = Reruns.

**BUFFALO RIVER PILOT PROJECT (CF#379)**  
**OIL AND GREASE IN SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE ug/g
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**BIN B**

379GLBR- 14	07OCT1:101SBO	1144.5 R
379GLBR- 21	07OCT12:531SBO	1537.6
379GLBR- 23	07OCT1:011SBO	2174.0
379GLBR- 65	09OCT9:542SB1O	2005.5
379GLBR- 66	09OCT10:152SB2O	2082.0
379GLBR- 67	09OCT10:352SB3O	1511.7
379GLBR- 174	25OCT12:153SB1O	2357.2
379GLBR- 176	25OCT2:153SB1O	1753.9 R
379GLBR- 181	25OCT2:254SB1O	560.4 U
379GLBR- 182	25OCT2:294SB1O	396.7 U
379GLBR- 183	25OCT2:284SB1O	464.2 U
379GLBR- 193	25OCT3:527SB1O	3770.7
379GLBR- 196	25OCT3:578SB1O	1898.7
379GLBR- 198	30OCT2:493SB2O	2071.2
379GLBR- 199 Rep 1	30OCT4:013SB2O	1634.5
379GLBR- 199 Rep 2	30OCT4:013SB2O	1306.0
379GLBR- 199 Rep 3	30OCT4:013SB2O	1610.2 R
379GLBR- 206	30OCT4:413SB2O	2148.9
379GLBR- 210	31OCT9:027SB2O	1231.0
379GLBR- 213	31OCT9:078SB2O	449.3
379GLBR- 220	31OCT9:264SB2O	203.7
379GLBR- 221	31OCT9:254SB2O	312.1
379GLBR- 222	31OCT9:244SB2O	144.0 U
379GLBR- 242	31OCT10:453SB3O	1192.5
379GLBR- 243	31OCT11:503SB3O	1232.5 R
379GLBR- 244	31OCT2:303SB3O	1549.8
379GLBR- 247	31OCT2:384SB3O	235.9 U R
379GLBR- 248	31OCT2:354SB3O	413.2 U
379GLBR- 249	31OCT2:364SB3O	520.0 U
379GLBR- 268	1NOV10:208SB3O	586.2 U
379GLBR- 269 Rep 1	1NOV10:157SB3O	1436.9
379GLBR- 269 Rep 2	1NOV10:157SB3O	1428.3
379GLBR- 269 Rep 3	1NOV10:157SB3O	1633.9

U = Analyte detected below detection limits.

R = Reruns.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
OIL AND GREASE IN SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE ug/g
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**BIN C**

379GLBR- 15	07OCT1:211SCO	2779.2
379GLBR- 18	07OCT1:331SCO	2180.1
379GLBR- 20	07OCT1:441SCO	1822.6
379GLBR- 73	09OCT4:562SC1O	3995.4
379GLBR- 74	09COT4:352SC2O	1761.2
379GLBR- 75	09OCT4:162SC3O	2398.5
379GLBR- 280	18NOV2:553SC1O	2374.9 R
379GLBR- 282	18NOV3:503SC1O	1845.4 R
379GLBR- 284	18NOV4:403SC1O	2105.6 R
379GLBR- 288	18NOV6:1510LC1O	6290.3
379GLBR- 302	19NOV8:594SC1O	527.6 U
379GLBR- 303	19NOV9:004SC1O	786.6 R
379GLBR- 304	19NOV9:014SC1O	371.5 U
379GLBR- 307	19NOV9:238SC1O	2538.7
379GLBR- 311	19NOV11:303SC2O	2729.1 R
379GLBR- 313	19NOV2:203SC2O	1663.3
379GLBR- 315	19NOV3:553SC2O	1641.1
379GLBR- 323	19NOV4:504SC2O	507.7 U
379GLBR- 324	19NOV4:504SC2O	471.6 U R
379GLBR- 325	19NOV4:504SC2O	549.3 U
379GLBR- 337	20NOV8:157SC2O	2416.0
379GLBR- 340	20NOV11:443SC3O	4897.3
379GLBR- 342	20NOV1:003SC3O	4759.8
379GLBR- 344	20NOV1:553SC3O	3107.3
379GLBR- 350	20NOV3:154SC3O	2695.4
379GLBR- 351	20NOV3:154SC3O	4609.4
379GLBR- 352	20NOV3:154SC3O	3549.0
379GLBR- 357	20NOV3:337SC3O	4388.9
379GLBR- 360 Rep 1	20NOV3:278SC3O	762.4
379GLBR- 360 Rep 2	20NOV3:278SC3O	505.3 U
379GLBR- 360 Rep 3	20NOV3:278SC3O	668.9 R

U = Analyte detected below detection limits.  
R = Reruns.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
OIL AND GREASE IN SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE ug/g
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**BIN D**

379GLBR- 19	07OCT2:041SDO	2105.9
379GLBR- 22	07OCT1:521SDO	1342.1
379GLBR- 24	07OCT2:122SDO	1671.9
379GLBR- 46	08OCT5:132SD3O	2347.2
379GLBR- 51	08OCT5:402SD1O	2416.3
379GLBR- 53	08OCT5:302SD2O	3038.9
379GLBR- 376	21NOV12:003SD1O	3197.3 R
379GLBR- 378	21NOV1:353SD1O	1054.8 R
379GLBR- 380	21NOV2:433SD1O	2921.6 R
379GLBR- 384 Rep 1	21NOV5:078SD1O	974.5
379GLBR- 384 Rep 2	21NOV5:078SD1O	748.9
379GLBR- 384 Rep 3	21NOV5:078SD1O	1172.9
379GLBR- 388	21NOV5:107SD1O	5368.3
379GLBR- 395	21NOV5:224SD1O	238.4 U
379GLBR- 396	21NOV5:224SD1O	790.9
379GLBR- 397	21NOV5:224SD1O	907.7

BLANK-1	0.0060
BLANK-2	0.0010
BLANK-3	0.0020
BLANK-4	0.0008
BLANK-5	0.0020
BLANK-6	0.0043
BLANK-7	0.0033
BLANK-8	0.0066
BLANK-9	0.0000

U = Analyte detected below detection limits.  
R = Reruns.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
OIL AND GREASE IN SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE ug/g
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**MATRIX SPIKE RESULTS**

**% RECOVERY**

379GLBR- 111 * *	22OCT5:563SA2O	2330.3	
379GLBR- 111 MATRIX SPIKE		3932.3	97%
379GLBR- 111 MATRIX SPIKE DUPLICATE		3369.5	82%
379GLBR- 182	25OCT2:294SB1O	610.0 U	
379GLBR- 182 MATRIX SPIKE		2190.7	158% *
379GLBR- 182 MATRIX SPIKE DUPLICATE		2159.0	153% *
379GLBR- 269 * *	1NOV10:157SB3O	1499.7	
379GLBR- 269 MATRIX SPIKE		2478.5	95%
379GLBR- 269 MATRIX SPIKE DUPLICATE		2533.9	91%
379GLBR- 360 * *	20NOV3:278SC3O	645.5	
379GLBR- 360 MATRIX SPIKE		1271.9	80%
379GLBR- 360 MATRIX SPIKE DUPLICATE		1590.8	99%
379GLBR- 384 * *	21NOV5:078SD1O	965.4	
379GLBR- 384 MATRIX SPIKE		2784.3	142% *
379GLBR- 384 MATRIX SPIKE DUPLICATE		2824.7	143% *

\*\* = Mean of replicated sample.

U = Analyte detected below detection limits.

\* = Outside control limits.

**BUFFALO RIVER PILOT PROJECT (CF#379)  
OIL AND GREASE IN SEDIMENT SAMPLES**

MSL CODE	SPONSOR ID	OIL & GREASE ug/g
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**REPLICATE ANALYSES**

379GLBR- 111 Rep 1	22OCT5:563SA2O	2867.7
379GLBR- 111 Rep 2	22OCT5:563SA2O	1839.4
379GLBR- 111 Rep 3	22OCT5:563SA2O	2283.9
	RSD %	22% *
379GLBR- 199 Rep 1	30OCT4:013SB2O	1634.5
379GLBR- 199 Rep 2	30OCT4:013SB2O	1306.0
379GLBR- 199 Rep 3	30OCT4:013SB2O	1610.2 R
	RSD %	12%
379GLBR- 269 Rep 1	1NOV10:157SB3O	1436.9
379GLBR- 269 Rep 2	1NOV10:157SB3O	1428.3
379GLBR- 269 Rep 3	1NOV10:157SB3O	1633.9
	RSD %	8%
379GLBR- 360 Rep 1	20NOV3:278SC3O	762.4
379GLBR- 360 Rep 2	20NOV3:278SC3O	610.0 U
379GLBR- 360 Rep 3	20NOV3:278SC3O	668.9 R
	RSD %	11%
379GLBR- 384 Rep 1	21NOV5:078SD1O	974.5
379GLBR- 384 Rep 2	21NOV5:078SD1O	748.9
379GLBR- 384 Rep 3	21NOV5:078SD1O	1172.9
	RSD %	22% *

U = Analyte detected below detection limits.

R = Reruns.

RSD% = Relative Standard Difference.

\* = Outside control limits.

BUFFALO RIVER PILOT PROJECT (CF #379)  
PAH ANALYSIS IN SEDIMENT SAMPLES  
(ANALYZED 10/91)

5/22/92

(Concentrations in ng/g)

MSL CODE	SPONSOR ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Fluorene	Phenan- threne	Anthra- cene	Fluoran- thene	Pyrene	Benzo(a)- Anthracene	Chrysene
379GLBR*15A	07OCT1:211SCO	47.16	34.74 U	52.92	75.80	633.88	157.45	1160.98	1017.31	542.02	675.73
379GLBR*15B	07OCT1:211SCO	38.15	34.60 U	53.73	82.98	682.24	178.79	1222.89	1063.39	578.78	691.00
379GLBR*15C	07OCT1:211SCO	35.59	32.13 U	51.81	83.49	627.06	159.06	1106.10	984.28	517.84	649.86
379GLBR*17	07OCT12:091SAO	37.64	44.42 U	58.76 U	59.52	522.95	110.44	1036.45	840.05	443.84	581.27
379GLBR*21	07OCT12:531SBO	34.39 U	44.27 U	58.55 U	51.40 U	522.85	118.72	1398.88	1137.65	631.32	756.99
379GLBR*22	07OCT1:521SDO	147.09	169.14	164.88	193.68	546.84	246.40	1027.16	859.27	561.82	665.26
Blank		16.79 U	21.61 U	28.58 U	25.09 U	15.56 U	18.44 U	11.83 U	12.24 U	10.57 U	9.36 U

STANDARD REFERENCE MATERIAL

SRM 1941	certified value
831.66	260.31 U
NC	NC
	344.29 U
	302.25 U
	482.72
	577.00
	222.14 U
	202.00
	988.04
	NC
	486.81
	550.00
	630.54
	NC

MATRIX SPIKE RESULTS

Amount Spiked											
379GLBR*22	07OCT1:521SDO	885.00	885.00	885.00	885.00	885.00	885.00	885.00	885.00	885.00	885.00
379GLBR*22 + Spike		147.09	169.14	164.88	193.68	546.84	246.40	1027.16	859.27	561.82	665.26
Amount Recovered		275.16	319.31	297.06	325.32	542.86	371.73	850.89	750.86	583.86	629.73
Percent Recovery		128.07	150.17	132.18	131.64	-3.98	125.33	-176.27	-108.41	22.04	-35.53
		14%*	17%*	15%*	15%*	0%*	14%*	-20%*	-12%*	2%*	-4%*
Amount Spiked											
379GLBR*22	07OCT1:521SDO	880.00	880.00	880.00	880.00	880.00	880.00	880.00	880.00	880.00	880.00
379GLBR*22 + Spike	DUPLICATE	147.09	169.14	164.88	193.68	546.84	246.40	1027.16	859.27	561.82	665.26
Amount Recovered		487.15	714.45	678.57	773.71	1177.08	921.38	1755.21	1565.81	1298.38	1332.46
Percent Recovery		340.06	545.31	513.69	580.03	630.24	674.98	728.05	706.54	736.56	667.2
		39%*	62%	58%	66%	72%	77%	83%	80%	84%	76%

U = Detected below detection limit  
\* = Recoveries outside of QC limits.  
NA = Not applicable.  
NC = Not certified.



BUFFALO RIVER PILOT PROJECT (CF #379)  
 PAH ANALYSIS IN SEDIMENT SAMPLES  
 (ANALYZED 10/91)

5/22/92

(Concentrations in ng/g)												
MSL CODE	SPONSOR ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Fluorene	Phenan- threne	Anthra- cene	Fluoran- thene	Pyrene	Benzo(a)- Anthracene	Chrysene	
REPLICATE ANALYSIS												
379GLBR*15A	07OCT1:21ISCO	47.16	34.74 U	52.92	75.80	633.88	157.45	1160.98	1017.31	542.02	675.73	
379GLBR*15B	07OCT1:21ISCO	38.15	34.60 U	53.73	82.98	682.24	178.79	1222.89	1063.39	578.78	691.00	
379GLBR*15C	07OCT1:21ISCO	35.59	32.13 U	51.81	83.49	627.06	159.06	1106.10	984.28	517.84	649.86	
	RSD%	15%	NA	2%	5%	5%	7%	5%	4%	6%	3%	

U - Detected below detection limit  
 \* - Recoveries outside of QC limits.  
 NA - Not applicable.  
 NC - Not certified.

BUFFALO RIVER PILOT PROJECT (CF #379)  
PAH ANALYSIS IN SEDIMENT SAMPLES  
(ANALYZED 10/91)

5/22/92

(Concentrations in ng/g)										% Surrogate Recovery	
MSL CODE	SPONSOR ID	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo(a) Pyrene	Indeno(1,2,3) Pyrene	Dibenzo(a,h) Anthracene	Benzo(g,h,i) Perylene	D8 Naph- thalene	D10 Acenaph- thalene	D12 Perylene	
379GLBR*15A	07OCT1:21ISCO	627.45	434.19	542.81	438.57	103.80	280.39	42%	48%	64%	
379GLBR*15B	07OCT1:21ISCO	644.37	454.13	560.96	453.05	114.59	287.02	36%	50%	67%	
379GLBR*15C	07OCT1:21ISCO	580.88	425.19	521.53	426.39	106.08	272.14	34%	50%	69%	
379GLBR*17	07OCT12:09ISAO	571.17	395.44	477.87	408.35	94.88	261.79	49%	57%	72%	
379GLBR*21	07OCT12:53ISBO	691.80	508.84	584.10	491.21	116.89	303.74	41%	57%	84%	
379GLBR*22	07OCT1:52ISDO	667.08	492.32	554.67	560.12	331.44	338.60	55%	62%	77%	
Blank		6.71 U	5.51 U	6.95 U	5.49 U	6.40 U	3.77 U	85%	85%	75%	
STANDARD REFERENCE MATERIAL											
SRM 1941	certified value	796.14	555.91	496.65	553.05	136.81	423.60	69%	74%	82%	
		780.00	444.00	670.00	569.00	NC	516.00	NA	NA	NA	
MATRIX SPIKE RESULTS											
Amount Spiked		885.00	885.00	885.00	885.00	885.00	885.00	NA	NA	NA	
379GLBR*22	07OCT1:52ISDO	667.08	492.32	554.67	560.12	331.44	338.60	55%	62%	77%	
379GLBR*22 + Spike		629.73	510.06	549.36	507.53	450.56	375.92	40%	43%	51%	
Amount Recovered		-37.35	17.74	-5.31	-52.59	119.12	37.32	NA	NA	NA	
Percent Recovery		-4%	2%	-1%	-6%	13%	4%	NA	NA	NA	
Amount Spiked		880.00	880.00	880.00	880.00	880.00	880.00	NA	NA	NA	
379GLBR*22	07OCT1:52ISDO	667.08	492.32	554.67	560.12	331.44	338.60	55%	62%	77%	
379GLBR*22 + Spike DUPLICATE		1355.48	1124.29	1204.26	1143.25	1096.43	837.02	52%	70%	93%	
Amount Recovered		688.4	631.97	649.59	583.13	764.99	498.42	NA	NA	NA	
Percent Recovery		78%	72%	74%	66%	87%	57%	NA	NA	NA	

U = Detected below detection limit  
\* = Recoveries outside of QC limits.  
NA = Not applicable.  
NC = Not certified.

BUFFALO RIVER PILOT PROJECT (CF #379)  
 PAH ANALYSIS IN SEDIMENT SAMPLES  
 (ANALYZED 10/91)

5/22/92

		(Concentrations in ng/g)						% Surrogate Recovery	
MSL CODE	SPONSOR ID	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo(a) Pyrene	Indeno(1,2,3) Pyrene	Dibenzo(a,h) Anthracene	Benzo(g,h,i) Perylene	D8 Naph- thalene	D10 Acenaph- thalene D12 Perylene
REPLICATE ANALYSIS									
379GLBR*15A	07OCT1:211SCO	627.45	434.19	542.81	438.57	103.80	280.39	42%	48%
379GLBR*15B	07OCT1:211SCO	644.37	454.13	560.96	453.05	114.59	287.02	36%*	50%
379GLBR*15C	07OCT1:211SCO	580.88	425.19	521.53	426.39	106.08	272.14	34%*	50%
	RSD%	5%	3%	4%	3%	5%	3%	NA	NA
									64%
									67%
									69%
									NA

U = Detected below detection limit  
 \* = Recoveries outside of QC limits.  
 NA = Not applicable.  
 NC = Not certified.

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene
<b>BIN A</b>								
379GLBR-97	21OCT6:1810LA10	134123	2361	15805 #	37080 #	86591 D	15925	8792
379GLBR-97 ~	21OCT6:2010LA10	106763	2289	14358	35122	104808	16862	10929
379GLBR-128	23OCT4:459LA20	39236	3069	12181 #	32308	110202	18271	16751
379GLBR-132	23OCT5:2010LA20	43851	4679	9611 #	33598	111982 D	26274	63737 D
379GLBR-160	25OCT10:1810LA30	24162	1751 #	3970 #	12339	55951 D	11386	37865 D
379GLBR-161	25OCT10:1910LA30	36540	3277 #	7019 #	22862	95140 D	20580	61455 D
379GLBR-164	25OCT9:549LA30	38413	9204	19760 #	64326	189784 D	39225	35555
379GLBR-204	30OCT4:206LA30	43 U	46 U	62 U	57 U	36 U	38 U	25 U
<b>BIN B</b>								
379GLBR-187	25OCT3:0810LB10	27983	5964	7333 #	22981	114021 D	30532	93778 D
379GLBR-226	31OCT10:119LB20	10474	125 #	926 #	2242	6422	239 #	1594
379GLBR-227	31OCT10:2310LB20	18845	2714	2273 #	7796	29804	6544	22030
379GLBR-252	31OCT3:119LB30	13540	202	2000 #	6608	32600 D	2759	14753 D
379GLBR-253	31OCT3:109LB30	11561	155	1896 #	5939	27611 D	1959	11711 D
379GLBR-256	31OCT3:0110LB30	14293	2084	2198 #	5784	29132 D	5948	18615 D
379GLBR-257	31OCT3:0010LB30	21054	3789	4594 #	12089	67219 D	14514	46912 D
<b>BIN C</b>								
379GLBR-286	18NOV5:559LC10	18313	2691	7161 #	23025	83440 D	14407	17479
379GLBR-326	19NOV5:509LC20	20689	3444	9437 #	28310	120301	22973	40218
379GLBR-331, Rep 1	19NOV5:0510LC20	5689	1707	2801 #	7669	44081	9728	33314
379GLBR-331, Rep 2	19NOV5:0510LC20	6110	1504	2271 #	5291	30611	6750	24642
379GLBR-331, Rep 3	19NOV5:0510LC20	5821	1294	1920	4018	23532	5246	19474
379GLBR-361	20NOV2:193LC30	60 B	27 U	36 U	33 U	110	23 U	80
379GLBR-366	20NOV2:429LC30	5181	1358	1697	4305	25498	7011	27038
379GLBR-370	20NOV3:0110LC30	9430	5368	8325	18667	91406 E	25842	81464 E

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>BIN A</b>								
379GLBR-97	21OCT6:1810LA10	33900 D	11082	33172 D	8935	21 U	13481 #	1451
379GLBR-97 ~	21OCT6:2010LA10	39571	11015	40731	9968	84 U	13156	1169
379GLBR-128	23OCT4:459LA20	49386	17602	39751	10321	153 U	11148	1104
379GLBR-132	23OCT5:2010LA20	64875 D	24528	50826	32041	51 U	16437	4268
379GLBR-160	25OCT10:1810LA30	37935 D	12637	28396 D	31 U	10940	8325	2174
379GLBR-161	25OCT10:1910LA30	62018 D	19820	45983 D	38	17677	13742	3389
379GLBR-164	25OCT9:549LA30	94035 D	38129	77380 D	32084	37 U	28968	5098
379GLBR-204	30OCT4:206LA30	26 U	30 U	25 U	24 U	17 U	24 U	22 U
<b>BIN B</b>								
379GLBR-187	25OCT3:0810LB10	71800 D	18980	30291 D	32 U	14581	7690	2579
379GLBR-226	31OCT10:119LB20	2328	603	1561	611	24 U	212 #	80
379GLBR-227	31OCT10:2310LB20	15136	5037	7478	6273	24 U	1585	614
379GLBR-252	31OCT3:119LB30	13079 D	3456	6596	3147	9 U	1020	266
379GLBR-253	31OCT3:109LB30	10912 D	2989	5688	2684	8	972	225
379GLBR-256	31OCT3:0110LB30	13914 D	4293	6558	3304	1580 #	1466	596
379GLBR-257	31OCT3:0010LB30	35469 D	11296	17116	8869	21 U	3867	1517
<b>BIN C</b>								
379GLBR-286	18NOV5:559LC10	39512 D	14062	30385 D	10454	22 U	8401	1369
379GLBR-326	19NOV5:509LC20	63531	23436	46161	18852	96 U	13019	2202
379GLBR-331, Rep 1	19NOV5:0510LC20	28145	7718	13840	6160	2558	3801	1260
379GLBR-331, Rep 2	19NOV5:0510LC20	20062	5570	9516	4514	2063	2425	925
379GLBR-331, Rep 3	19NOV5:0510LC20	15593	4340	7121	3824	1461	1911	716
379GLBR-361	20NOV2:193LC30	68	18 U	31	17 #	10 U	14 U	13 U
379GLBR-366	20NOV2:429LC30	21804	6941	10309	9689	69 U	2958	1128
379GLBR-370	20NOV3:0110LC30	62530 E	17229	24776	14282	5931 #	6370	3062

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

(Concentrations in ng/L)				% Surrogate Recovery		
MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	D8-Naphthalene	D-10 Ace-naphthalene	D12-Perylene
<b>BIN A</b>						
379GLBR-97	21OCT6:1810LA10	3323 #	6622	23% •	31% •	27% •
379GLBR-97 •	21OCT6:2010LA10	3229	7315	20% •	29% •	33% •
379GLBR-128	23OCT4:459LA20	2086 #	7981	12% •	22% •	23% •
379GLBR-132	23OCT5:2010LA20	3406 #	7772	16% •	21% •	24% •
379GLBR-160	25OCT10:1810LA30	1960 #	4423	43% •	53% •	55% •
379GLBR-161	25OCT10:1910LA30	3413 #	7099	41% •	50% •	48% •
379GLBR-164	25OCT9:549LA30	5963 #	16790	9% •	28% •	34% •
379GLBR-204	30OCT4:206LA30	21 U	27 #	82% •	81% •	83% •
<b>BIN B</b>						
379GLBR-187	25OCT3:0810LB10	1616 #	3648	68% •	61% •	66% •
379GLBR-226	31OCT10:119LB20	68 #	208	16% •	24% •	23% •
379GLBR-227	31OCT10:2310LB20	211	602	24% •	31% •	31% •
379GLBR-252	31OCT3:119LB30	188 #	523	24% •	33% •	29% •
379GLBR-253	31OCT3:109LB30	190 #	528	22% •	32% •	30% •
379GLBR-256	31OCT3:0110LB30	213	610	21% •	30% •	32% •
379GLBR-257	31OCT3:0010LB30	543	1525	21% •	28% •	28% •
<b>BIN C</b>						
379GLBR-286	18NOV5:559LC10	1702 #	5736	16% •	31% •	33% •
379GLBR-326	19NOV5:509LC20	2373 #	8312	12% •	22% •	27% •
379GLBR-331, Rep 1	19NOV5:0510LC20	688 #	1873	20% •	29% •	36% •
379GLBR-331, Rep 2	19NOV5:0510LC20	428 #	1223	27% •	33% •	37% •
379GLBR-331, Rep 3	19NOV5:0510LC20	261 #	882	24% •	33% •	34% •
379GLBR-361	20NOV2:193LC30	12 U	11 B	28% •	31% •	38% •
379GLBR-366	20NOV2:429LC30	426	1278	18% •	30% •	29% •
379GLBR-370	20NOV3:0110LC30	997	3062	21% •	31% •	32% •

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene	Fluoranthene
<b>BIN D</b>								
379GLBR-398	21NOV3:2610LD10	5543	819	1796 #	4039	19280	3992	15163
379GLBR-404	21NOV4:169LD10	4211	2106	2095 #	7287	36954	11917	39774
<b>DILUTION WATER</b>								
379GLBR-275	6NOV12:003LO	30 B	19 U	26 U	24 U	34	16 U	32
BLANK		85	53 U	70 U	65 U	41 U	44 U	29 U
<b>MATRIX SPIKE RESULTS (1)</b>								
Amount Spiked		6410	6410	6410	6410	6410	6410	6410
379GLBR-132	23OCT5:2010LA20	43851	4679	9611 #	33598	111982 D	26274	63737 D
379GLBR-132 + Spike		23639	3869	6324 #	20300	68000	16648	39157
Amount Recovered		-20212	-810	-3287	-13298	-43982	-9626	-24580
Percent Recovery		-315% *	-13% *	-51% *	-207% *	-686% *	-150% *	-383% *
Amount Spiked		7143	7143	7143	7143	7143	7143	7143
379GLBR-132	23OCT5:2010LA20	43851	4679	9611 #	33598	111982 D	26274	63737 D
379GLBR-132 + Spike	DUPLICATE	48960	6735	11133	34056	126052 E	28647	71703
Amount Recovered		5109	2056	1522	458	14070	2373	7966
Percent Recovery		72%	29% *	21% *	6% *	197% *	33% *	112%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]-anthracene	Chrysene	Benzo(b)-Fluoranthene	Benzo(k)-Fluoranthene	Benzo[a]-pyrene	Indeno [1,2,3-cd]-pyrene
<b>BIN D</b>								
379GLBR-398	21NOV3:2610LD10	12281	3051	5001	4089	57	1097	537
379GLBR-404	21NOV4:169LD10	30273	9684	14186	7395	3751	3365	1217
<b>DILUTION WATER</b>								
379GLB R-275	6NOV12:003LO	65	13 U	19	10 U	7 U	10 U	9 U
BLANK		30 U	34 U	29 U	27 U	20 U	27 U	25 U
<b>MATRIX SPIKE RESULTS</b>								
Amount Spiked		6410	6410	6410	6410	6410	6410	6410
379GLBR-132	23OCT5:2010LA20	64875 D	24528	50826	32041	51 U	16437	4268
379GLBR-132 + Spike		40044	154397	30532	13969	5378	10568	3727
Amount Recovered		-24831	129869	-20294	-18072	5378	-5869	-541
Percent Recovery		-387% *	2026% *	-317% *	-282% *	84%	-92% *	-8% *
Amount Spiked		7143	7143	7143	7143	7143	7143	7143
379GLBR-132	23OCT5:2010LA20	64875 D	24528	50826	32041	51 U	16437	4268
379GLBR-132 + Spike	DUPLICATE	71305	25861	53761	23306	10431	16661	6394
Amount Recovered		6430	1333	2935	-8735	10431	224	2126
Percent Recovery		90%	19% *	41%	-122% *	146% *	3% *	30% *



**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

				% Surrogate Recovery			
MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	D8-Naphthalene	D-10 Ace-naphthalene	D12-Perylene	
<div>BIN D</div>							
379GLBR-398	21NOV3:2610LD10	210	521	26% *	29% *	24% *	
379GLBR-404	21NOV4:169LD10	386	1201	8% *	15% *	13% *	
<div>DILUTION WATER</div>							
379GLBR-275	6NOV12:003LO	9 U	10 B	21% *	28% *	38% *	
BLANK		24 U	33	61%	64%	67%	
MATRIX SPIKE RESULTS							
Amount Spiked		6410	6410	NA	NA	NA	
379GLBR-132	23OCT5:2010LA20	3406 #	7772	16% *	21% *	24% *	
379GLBR-132 + Spike		3654	5550	14% *	21% *	24% *	
Amount Recovered		248	-2222	NA	NA	NA	
Percent Recovery		4% *	-35% *	NA	NA	NA	
Amount Spiked		7143	7143	NA	NA	NA	
379GLBR-132	23OCT5:2010LA20	3406 #	7772	16% *	21% *	24% *	
379GLBR-132 + Spike	DUPLICATE	5462	9791	18% *	23% *	25% *	
Amount Recovered		2056	2019	NA	NA	NA	
Percent Recovery		29% *	28% *	NA	NA	NA	

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Flourene	Phenan- threne	Anthra- cene	Fluoran- thene
<b>REPLICATE ANALYSIS</b>								
379GLBR-331, Rep 1	19NOV5:0510LC20	5689	1707	2801 #	7669	44081	9728	33314
379GLBR-331, Rep 2	19NOV5:0510LC20	6110	1504	2271 #	5291	30611	6750	24642
379GLBR-331, Rep 3	19NOV5:0510LC20	5821	1294	1920	4018	23532	5246	19474
	<b>RSD %</b>	<b>4%</b>	<b>14%</b>	<b>19%</b>	<b>33% ..</b>	<b>32% ..</b>	<b>32% ..</b>	<b>27% ..</b>

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>REPLICATE ANALYSIS</b>								
379GLBR-331, Rep 1	19NOV5:0510LC2O	28145	7718	13840	6160	2558	3801	1260
379GLBR-331, Rep 2	19NOV5:0510LC2O	20062	5570	9516	4514	2063	2425	925
379GLBR-331, Rep 3	19NOV5:0510LC2O	15593	4340	7121	3824	1461	1911	716
	<b>RSD %</b>	<b>30% ..</b>	<b>29% ..</b>	<b>34% ..</b>	<b>25% ..</b>	<b>27% ..</b>	<b>36% ..</b>	<b>28% ..</b>

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN WATER SAMPLES**

*(Concentrations in ng/L)*

MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene		Benzo[ghi]perylene	% Surrogate Recovery			
					D8-Naphthalene	D-10 Ace-naphthalene	D12-Perylene	
REPLICATE ANALYSIS								
379GLBR-331, Rep 1	19NOV5:0510LC20	688 #	1873		20% *	29% *	36% *	
379GLBR-331, Rep 2	19NOV5:0510LC20	428 #	1223		27% *	33% *	37% *	
379GLBR-331, Rep 3	19NOV5:0510LC20	261 #	882		24% *	33% *	34% *	
	RSD %	47% **	38% **		NA	NA	NA	

- U = Detected at or below detection limit.  
 B = Naphthalene and Benzo(ghi)perylene were detected at levels near detection limits.  
 ~ = Field replicate.  
 # = Indicates confirming ion out of specification.  
 D = 10:1 sample dilution.  
 E = Indicates value outside of calibration.  
 RSD % = Relative standard deviation.  
 \* = Recoveries exceed laboratory control limits (40-120%).  
 \*\* = Value exceeds precision goal of 20%.

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Flourene	Dibenzo- thiophene	Phenan- threne	Anthra- cene	Fluoran- thene
<b>BIN A</b>									
379GLBR-13	07OCT12:341SAO	73	34	35	64	34	437	96	1101
379GLBR-16	07OCT2:401SAO	62	34	36	64	43	475	95	1160
379GLBR-57	09OCT11:502SA3O	102	42	52	80	42	552	120	1363
379GLBR-58	09OCT11:382SA2O	66	46	93	138	67	948	180	1731
379GLBR-59	09OCT11:282SA1O	67	31	39	69	39	473	104	1169
379GLBR-104	22OCT11:317SA1O	573	29	162 #	560	1884	2716	373	2271
379GLBR-107	22OCT11:378SA1O	126	16	25 #	102	167	593	141	1448
379GLBR-108	22OCT4:203SA2O	35	35	43	81	44	608	118	1326
379GLBR-111	22OCT5:563SA2O	158	88	109	160	82	1166	230	2726
379GLBR-111-R	22OCT5:563SA2O	36 U	44	53 U	46	32	446	93	978
379GLBR-113	23OCT1:303SA2O	65	33	40	59	38	462	91	1114
379GLBR-122	23OCT4:284SA2O	24	4 U	7 U	5 U	4 U	21	4 U	14
379GLBR-122-R	23OCT4:284SA2O	45	5 U	8 U	6 U	15	38	4 U	20
379GLBR-123	23OCT4:274SA2O	39	7 U	13 U	10 U	9	29	6 U	21
379GLBR-124	23OCT4:294SA2O	47	4 U	7 U	6	8	34	4 U	26
379GLBR-124-R	23OCT4:294SA2O	61	6 U	11 U	7 U	21	64	7	59
379GLBR-136	24OCT10:457SA2O	221	13	70	198	476	832	137	566
379GLBR-139	24OCT10:508SA2O	275	14 U	31 #	97	218	405	65	250
379GLBR-141	24OCT2:203SA3O	23 U	49	38	56	36	543	85	1162
379GLBR-143	24OCT5:503SA3O	44	34	45	62	41	605	82	1308
379GLBR-145	24OCT6:203SA3O	60	29	46	67	39	453	80	1060
379GLBR-151	25OCT9:224SA3O	25	6 U	10 U	7 U	9	101	17	198
379GLBR-152	25OCT9:224SA3O	27	8 U	14 U	12	15	180	24	400
379GLBR-153	25OCT9:214SA3O	9	7 U	12 U	10 U	11	109	17	263
379GLBR-166	25OCT9:307SA3O	36	14	18	48	72	540	110	1890
379GLBR-167	25OCT9:368SA3O	33	8 U	15 U	18	28	187	38	584

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>BIN A</b>								
379GLBR-13	07OCT12:341SAO	792	409	553	612	485	497	370
379GLBR-16	07OCT2:401SAO	827	394	583	677	479	501	391
379GLBR-57	09OCT11:502SA3O	962	483	659	790	521	519	449
379GLBR-58	09OCT11:382SA2O	1220	638	767	987	658	634	545
379GLBR-59	09OCT11:282SA1O	845	410	569	663	458	471	376
379GLBR-104	22OCT11:317SA1O	3071	1933	3326	2920	4 U	1821	657
379GLBR-107	22OCT11:378SA1O	1114	888	1277	1740 #	930 #	972	749
379GLBR-108	22OCT4:203SA2O	937	489	609	772	537	503	433
379GLBR-111	22OCT5:563SA2O	1863	918	1335	1528	1022	845	816
379GLBR-111-R	22OCT5:563SA2O	737	339	487	507	373	372	294
379GLBR-113	23OCT1:303SA2O	805	433	585	680	476	447	372
379GLBR-122	23OCT4:284SA2O	6	3 U	8	4	2 U	3 U	2 U
379GLBR-122-R	23OCT4:284SA2O	18	8	17	19	2 U	4	3
379GLBR-123	23OCT4:274SA2O	15	9	15	11 #	5 #	4 U	3
379GLBR-124	23OCT4:294SA2O	19	10	21	19	2 U	3 U	2
379GLBR-124-R	23OCT4:294SA2O	46	21	41	52	3 U	10	7
379GLBR-136	24OCT10:457SA2O	620	381	610	744	340 #	337	302
379GLBR-139	24OCT10:508SA2O	246	139	263	361	8 U	119	71
379GLBR-141	24OCT2:203SA3O	788	360	589	676	496	368	369
379GLBR-143	24OCT5:503SA3O	906	429	703	828	611	408	443
379GLBR-145	24OCT6:203SA3O	777	367	521	629	477	459	362
379GLBR-151	25OCT9:224SA3O	136	73	124	200	2 U	46	33
379GLBR-152	25OCT9:224SA3O	248	148	267	326	218	96	151
379GLBR-153	25OCT9:214SA3O	173	104	166	206	135	95	90
379GLBR-166	25OCT9:307SA3O	1273	812	1155	1289 #	747 #	614	428
379GLBR-167	25OCT9:368SA3O	380	350	536	783	424	306	240

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

				% Surrogate Recovery			
MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	d8 Naphthalene	d10 Acenaphthene	Fluorene	D10-Dibenzo(a,h)Anthracene
BIN A							
379GLBR-13	07OCT12:341SAO	141	358	77%	73%	99%	131%
379GLBR-16	07OCT2:401SAO	150	368	62%	66%	96%	132%
379GLBR-57	09OCT11:502SA3O	182	397	90%	72%	103%	129%
379GLBR-58	09OCT11:382SA2O	200	480	81%	48%	97%	111%
379GLBR-59	09OCT11:282SA1O	151	316	54%	54%	86%	116%
379GLBR-104	22OCT11:317SA1O	501 #	1269	52%	25%	88%	97%
379GLBR-107	22OCT11:378SA1O	335	798	64%	34%	87%	102%
379GLBR-108	22OCT4:203SA2O	124	385	39%	26%	79%	94%
379GLBR-111	22OCT5:563SA2O	330	582	42%	36%	75%	89%
379GLBR-111-R	22OCT5:563SA2O	74	294	33%	72%	65%	91%
379GLBR-113	23OCT1:303SA2O	150	364	43%	41%	75%	93%
379GLBR-122	23OCT4:284SA2O	3 U	2 U	24%	9%	52%	4%
379GLBR-122-R	23OCT4:284SA2O	2	3	67%	73%	75%	21%
379GLBR-123	23OCT4:274SA2O	3 U	4	68%	35%	80%	15%
379GLBR-124	23OCT4:294SA2O	3 U	5	39%	26%	63%	25%
379GLBR-124-R	23OCT4:294SA2O	4	7	71%	80%	73%	34%
379GLBR-136	24OCT10:457SA2O	140	314	50%	45%	74%	85%
379GLBR-139	24OCT10:508SA2O	42	89	80%	71%	83%	82%
379GLBR-141	24OCT2:203SA3O	123	345	4%	48%	69%	96%
379GLBR-143	24OCT5:503SA3O	163	399	53%	50%	82%	110%
379GLBR-145	24OCT6:203SA3O	97	363	98%	89%	97%	111%
379GLBR-151	25OCT9:224SA3O	17	30	74%	80%	77%	41%
379GLBR-152	25OCT9:224SA3O	67	135	67%	54%	81%	90%
379GLBR-153	25OCT9:214SA3O	44	85	17%	54%	72%	76%
379GLBR-166	25OCT9:307SA3O	162	441	59%	68%	78%	99%
379GLBR-167	25OCT9:368SA3O	96	239	61%	62%	79%	88%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Flourene	Dibenzo- thiophene	Phenan- threne	Anthra- cene	Fluoran- thene
<b>BIN B</b>									
379GLBR-14	07OCT1:101SBO	79	57	34	64	38	425	108	1182
379GLBR-23	07OCT1:011SBO	109	46	41	87	49	593	123	1447
379GLBR-65	09OCT9:542SB10	53	33	55	87	55	550	117	1172
379GLBR-66	09OCT10:152SB20	135	38	62	91	56	640	148	1536
379GLBR-67	09OCT10:352SB30	115	37	64	79	46	562	125	1379
379GLBR-174	25OCT12:153SB10	76	32	49	68	42	425	81	957
379GLBR-176	25OCT12:153SB10	40	32	49	74	47	564	106	1245
379GLBR-181	25OCT12:254SB10	23	8	10 U	27	144	338	38	418
379GLBR-182, Rep 1	25OCT12:294SB10	27	7 U	12 U	16	11	146	18	274
379GLBR-182, Rep 2	25OCT12:294SB10	22	11 U	69 #	21	18	191	35	356
379GLBR-182, Rep 3	25OCT12:294SB10	26	11 U	72 #	16	14	143	29	277
379GLBR-183	25OCT12:284SB10	20	10 U	58 #	12 U	10	115	19	209
379GLBR-193	25OCT13:527SB10	65	17	30 #	111	116	887	191	2301 D
379GLBR-196	25OCT13:578SB10	50	18	28	101	88	942	203	2599 D
379GLBR-198	30OCT2:493SB20	44	32	59 #	105	55	727	152	1239
379GLBR-199, Rep 1	20OCT3:573SB20	62	31	52	74	48	503	91	1123
379GLBR-199, Rep 2	20OCT3:573SB20	49	29	42	59	40	424	81	931
379GLBR-199, Rep 3	20OCT3:573SB20	59	23	40	61	44	472	93	892
379GLBR-206	30OCT4:413SB20	63	43	268 #	222	127	2131	429	2920
379GLBR-210	31OCT9:027SB20	375	24	46 #	142	142	750	146	1173
379GLBR-213	31OCT9:078SB20	161	8 U	15 U	42	53	287	47	420
379GLBR-220	31OCT9:264SB20	19	17 U	28 U	22 U	14 U	12 U	14 U	10 U
379GLBR-220-R	31OCT9:264SB20	30	6 U	11 U	8 U	6 U	19	5 U	7
379GLBR-221	31OCT9:254SB20	18	10 U	16 U	13 U	8 U	10	8 U	6 U
379GLBR-221-R	31OCT9:254SB20	28	4 U	8 U	6 U	5 U	15	4 U	4
379GLBR-222	31OCT9:244SB20	24 U	20 U	36 U	30 U	20 U	17 U	19 U	17 U
379GLBR-222-R	31OCT9:244SB20	44	9 U	15 U	11 U	8 U	19	7 U	6 U
379GLBR-242	31OCT10:453SB30	35	29	49	56	39	499	96	1005
379GLBR-243	31OCT11:503SB30	60	34	36	56	37	426	87	1032
379GLBR-244	31OCT12:303SB30	47	32	103 #	70	49	614	128	1197
379GLBR-247	31OCT12:384SB30	21	12 U	22 #	14 U	16	90	13	166



**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>BIN B</b>								
379GLBR-14	07OCT1:101SBO	887	455	630	789	546	654	544
379GLBR-23	07OCT1:011SBO	1021	491	695	797	540	564	444
379GLBR-65	09OCT9:542SB10	868	451	583	723	511	519	413
379GLBR-66	09OCT10:152SB20	1128	571	742	874	581	655	483
379GLBR-67	09OCT10:352SB30	999	520	695	791	558	585	452
379GLBR-174	25OCT12:153SB10	720	347	480	596	416	453	337
379GLBR-176	25OCT2:153SB10	872	561	959	859	622	397	418
379GLBR-181	25OCT2:254SB10	409	209	371	349	189 #	196	168
379GLBR-182, Rep 1	25OCT2:294SB10	173	98	187	243	147	29	104
379GLBR-182, Rep 2	25OCT2:294SB10	254	145	217	239	154	97	107
379GLBR-182, Rep 3	25OCT2:294SB10	196	122	195	231	137	94	100
379GLBR-183	25OCT2:284SB10	139	87	151	176	111	58	86
379GLBR-193	25OCT3:527SB10	1616 D	1162	1584 D	1873	969 #	836	538
379GLBR-196	25OCT3:578SB10	1848	1001	1354	1386 #	858 #	638	433
379GLBR-198	30OCT2:493SB20	933	472	614	683	515	510	395
379GLBR-199, Rep 1	20OCT3:573SB20	833	376	517	631	457	447	355
379GLBR-199, Rep 2	20OCT3:573SB20	696	322	433	499	378	383	295
379GLBR-199, Rep 3	20OCT3:573SB20	649	298	410	470	335	343	268
379GLBR-206	30OCT4:413SB20	2310	1197	1371	1364	1075	1058	771
379GLBR-210	31OCT9:027SB20	775	620	863	1340	738	615	613
379GLBR-213	31OCT9:078SB20	258	230	369	501	206 #	113	83
379GLBR-220	31OCT9:264SB20	9 U	8 U	7 U	7 U	6 U	8 U	5 U
379GLBR-220-R	31OCT9:264SB20	7	4 U	5	4 U	3 U	3 U	2 U
379GLBR-221	31OCT9:254SB20	5 U	5 U	4 U	4 U	4 U	5 U	3 U
379GLBR-221-R	31OCT9:254SB20	4	3 U	3	3 U	2 U	3 U	2 U
379GLBR-222	31OCT9:244SB20	15 U	14 U	13 U	15 U	13 U	16 U	10 U
379GLBR-222-R	31OCT9:244SB20	5 U	5 U	5 U	5 U	4 U	5 U	4 U
379GLBR-242	31OCT10:453SB30	761	359	508	580	441	434	340
379GLBR-243	31OCT11:503SB30	757	373	500	605	441	423	347
379GLBR-244	31OCT2:303SB30	905	463	603	695	482	512	378
379GLBR-247	31OCT2:384SB30	109	73	132	253	4 U	53	67

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	BIN B	Dibenzo[a,h]-		Benzo[ghi] perylene	% Surrogate Recovery			
			anthracene			Fluorene		D10- Fluorene	D14 Dibenzo- (a,h)Anthracene
			d8 Naph- thalene	d10 Acena- phtene					
379GLBR-14	07OCT1:101SBO	307	642	75%	69%	90%	122%		
379GLBR-23	07OCT1:011SBO	171	401	91%	78%	109%	145%		
379GLBR-65	09OCT9:542SB10	161	386	66%	45%	90%	104%		
379GLBR-66	09OCT10:152SB20	195	422	83%	64%	107%	132%		
379GLBR-67	09OCT10:352SB30	182	388	84%	64%	100%	123%		
379GLBR-174	25OCT12:153SB10	118	340	78%	65%	79%	86%		
379GLBR-176	25OCT2:153SB10	161	383	54%	54%	79%	102%		
379GLBR-181	25OCT2:254SB10	73	189	52%	65%	78%	93%		
379GLBR-182, Rep 1	25OCT2:294SB10	48	90	62%	45%	78%	89%		
379GLBR-182, Rep 2	25OCT2:294SB10	51	99	47%	43%	64%	73%		
379GLBR-182, Rep 3	25OCT2:294SB10	48	96	66%	62%	78%	87%		
379GLBR-183	25OCT2:284SB10	40	86	47%	43%	63%	73%		
379GLBR-193	25OCT3:527SB10	231	569	66%	58%	85%	101%		
379GLBR-196	25OCT3:578SB10	178	444	69%	56%	80%	93%		
379GLBR-198	30OCT2:493SB20	113	379	46%	51%	72%	91%		
379GLBR-199, Rep 1	20OCT3:573SB20	130	353	87%	73%	94%	105%		
379GLBR-199, Rep 2	20OCT3:573SB20	106	291	74%	62%	78%	87%		
379GLBR-199, Rep 3	20OCT3:573SB20	96	253	70%	61%	78%	89%		
379GLBR-206	30OCT4:413SB20	311	709	54%	50%	75%	95%		
379GLBR-210	31OCT9:027SB20	255	618	87%	76%	96%	117%		
379GLBR-213	31OCT9:078SB20	53	67	80%	65%	88%	55%		
379GLBR-220	31OCT9:264SB20	6 U	5 U	47%	23%	60%	5%		
379GLBR-220-R	31OCT9:264SB20	3 U	3 U	63%	66%	65%	3%		
379GLBR-221	31OCT9:254SB20	3 U	3 U	45%	39%	57%	7%		
379GLBR-221-R	31OCT9:254SB20	2 U	2 U	73%	74%	77%	3%		
379GLBR-222	31OCT9:244SB20	12 U	11 U	10%	40%	71%	5%		
379GLBR-222-R	31OCT9:244SB20	4 U	4 U	77%	80%	75%	5%		
379GLBR-242	31OCT10:453SB30	92	326	40%	39%	66%	86%		
379GLBR-243	31OCT11:503SB30	129	324	74%	64%	85%	101%		
379GLBR-244	31OCT2:303SB30	109	374	49%	48%	71%	92%		
379GLBR-247	31OCT2:384SB30	32	64	50%	39%	55%	63%		

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Dibenzothiophene	Phenanthrene	Anthracene	Fluoranthene
<b>BIN B</b>									
379GLBR-247-R	31OCT2:384SB30	45 U	39 U	68 U	9 U	21	141	18	171
379GLBR-248	31OCT2:354SB30	21	7 U	13 U	10 U	18	101	11	191
379GLBR-249	31OCT2:364SB30	26	15 U	26 U	19 U	16	96	13	196
379GLBR-268	1NOV10:208SB30	81	8 U	15 U	34	74	364	60	763
379GLBR-268-R	1NOV10:208SB30	109	20 U	36 U	38	76	450	85	771
379GLBR-269	1NOV10:157SB30	84	14	22	61	137	612	107	1253
<b>BIN C</b>									
379GLBR-18, Rep 1	07OCT1:331SCO	147	66	92	147	105	923	264	1938
379GLBR-18, Rep 2	07OCT1:331SCO	94	41	67	95	75	615	167	1343
379GLBR-18, Rep 3	07OCT1:331SCO	125	53	91	131	100	835	237	1793
379GLBR-20	07OCT1:441SCO	105	34	59	101	77	570	143	1245
379GLBR-73	09OCT4:562SC10	101	53	97	153	105	875	193	1552
379GLBR-74	09OCT4:352SC20	94	41	78	105	78	661	164	1397
379GLBR-75	09OCT4:162SC30	121	43	69	105	73	659	151	1444
379GLBR-280	18NOV2:553SC10	67	44	65 #	91	69	613	130	1139
379GLBR-282	18NOV3:503SC10	68	32	72	108	79	697	136	1328
379GLBR-284	18NOV4:403SC10	113	60	134	173	134	1155	244	1975
379GLBR-288	18NOV6:1510LC10	115	41	57	128	216	1197	277	4360
379GLBR-302	19NOV8:594SC10	33	7 U	13 U	11 U	16	98	15	212
379GLBR-303	19NOV9:004SC10	23	11 U	18 U	13 U	22	125	22	286
379GLBR-304	19NOV9:014SC10	27	11 U	21 #	13 U	19	98	16	211
379GLBR-307	19NOV9:238SC10	281	13	41 #	121	304	799	104	1069
379GLBR-311	19NOV11:303SC20	109	39	81	118	87	684	144	1334 D
379GLBR-313	19NOV2:203SC20	78	48	87	111	88	713	153	1295
379GLBR-315	19NOV3:553SC20	34	29	54	85	67	550	133	1080
379GLBR-323, Rep 1	19NOV4:504SC20	36	7 U	12 U	10 U	24	128	20	267
379GLBR-323, Rep 2	19NOV4:504SC20	31	10	10 U	13	28	174	28	379
379GLBR-323, Rep 3	19NOV4:504SC20	31	7 U	11 U	9 U	22	125	20	265
379GLBR-324	19NOV4:504SC20	34	12 U	21 U	17 U	56	142	19	229

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>BIN B</b>								
379GLBR-247-R	31OCT2:384SB3O	121	87	156	262	3 U	60	59
379GLBR-248	31OCT2:354SB3O	116	70	131	163	97	43	62
379GLBR-249	31OCT2:364SB3O	129	89	160	175	106	59	70
379GLBR-268	1NOV10:208SB3O	475	360	562	806	422 #	281	249
379GLBR-268-R	1NOV10:208SB3O	507	391	637	1217	8 U	286	254
379GLBR-269	1NOV10:157SB3O	822	700	1031	1639	904 #	719	595
<b>BIN C</b>								
379GLBR-18, Rep 1	07OCT1:331SCO	1534	785	957	1039	777	912	621
379GLBR-18, Rep 2	07OCT1:331SCO	1077	528	664	766	513	601	438
379GLBR-18, Rep 3	07OCT1:331SCO	1436	747	893	1033	695	852	583
379GLBR-20	07OCT1:441SCO	972	477	634	700	475	522	385
379GLBR-73	09OCT4:562SC1O	1211	615	762	921	634	639	515
379GLBR-74	09OCT4:352SC2O	1073	533	692	761	537	598	436
379GLBR-75	09OCT4:162SC3O	1098	541	704	794	560	608	457
379GLBR-280	18NOV2:553SC1O	939	467	634	696	537	577	407
379GLBR-282	18NOV3:503SC1O	1022	470	601	715	503	556	396
379GLBR-284	18NOV4:403SC1O	1597	782	999	1054	826	872	614
379GLBR-288	18NOV6:1510LC1O	3446	1879	2446	4861	10 U	1664 #	1095
379GLBR-302	19NOV8:594SC1O	145	101	184	383	4 U	97	105
379GLBR-303	19NOV9:004SC1O	205	157	264	322	204	148	164
379GLBR-304	19NOV9:014SC1O	152	114	199	245	140	102	117
379GLBR-307	19NOV9:238SC1O	963	633	1175	1566	4 U	605	302
379GLBR-311	19NOV11:303SC2O	1057 D	473	655	764	542	622	414
379GLBR-313	19NOV2:203SC2O	1058	494	682	755	539	590	413
379GLBR-315	19NOV3:553SC2O	843	407	526	618	424	465	337
379GLBR-323, Rep 1	19NOV4:504SC2O	189	131	238	484	4 U	131	137
379GLBR-323, Rep 2	19NOV4:504SC2O	277	178	296	361	229	189	180
379GLBR-323, Rep 3	19NOV4:504SC2O	180	126	240	308	174	123	141
379GLBR-324	19NOV4:504SC2O	156	113	209	245	145	82	125

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	% Surrogate Recovery			
				d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo[a,h]Anthracene

<b>BIN B</b>							
379GLBR-247-R	31OCT2:384SB30	30	62	1% *	131% *	52%	67%
379GLBR-248	31OCT2:354SB30	31	54	55%	59%	82%	79%
379GLBR-249	31OCT2:364SB30	35	65	61%	41%	74%	74%
379GLBR-268	1NOV10:208SB30	108	224	61%	31% *	71%	64%
379GLBR-268-R	1NOV10:208SB30	116	261	76%	82%	78%	78%
379GLBR-269	1NOV10:157SB30	249	571	70%	65%	91%	111%

<b>BIN C</b>							
379GLBR-18, Rep 1	07OCT1:331SCO	251	602	64%	59%	89%	123% *
379GLBR-18, Rep 2	07OCT1:331SCO	179	388	57%	61%	84%	121% *
379GLBR-18, Rep 3	07OCT1:331SCO	241	505	72%	68%	96%	133% *
379GLBR-20	07OCT1:441SCO	161	345	80%	69%	100%	133% *
379GLBR-73	09OCT4:562SC10	195	475	84%	46%	104%	116%
379GLBR-74	09OCT4:352SC20	176	397	78%	76%	101%	129% *
379GLBR-75	09OCT4:162SC30	183	415	87%	61%	102%	122% *
379GLBR-280	18NOV2:553SC10	150	394	59%	48%	79%	93%
379GLBR-282	18NOV3:503SC10	156	368	54%	61%	91%	106%
379GLBR-284	18NOV4:403SC10	236	585	65%	30% *	87%	89%
379GLBR-288	18NOV6:1510LC10	481	1154	69%	63%	85%	124% *
379GLBR-302	19NOV8:594SC10	48	104	90%	80%	99%	104%
379GLBR-303	19NOV9:004SC10	76	165	62%	43%	79%	91%
379GLBR-304	19NOV9:014SC10	52	117	54%	32% *	71%	78%
379GLBR-307	19NOV9:238SC10	170	456	58%	70%	82%	100%
379GLBR-311	19NOV11:303SC20	159	407	96%	89%	111%	130% *
379GLBR-313	19NOV2:203SC20	152	397	65%	54%	85%	97%
379GLBR-315	19NOV3:553SC20	117	336	37% *	46%	80%	101%
379GLBR-323, Rep 1	19NOV4:504SC20	54	144	86%	77%	89%	96%
379GLBR-323, Rep 2	19NOV4:504SC20	71	189	84%	76%	91%	101%
379GLBR-323, Rep 3	19NOV4:504SC20	55	146	83%	74%	88%	99%
379GLBR-324	19NOV4:504SC20	55	128	52%	48%	74%	87%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Flourene	Dibenzo- thiophene	Phenan- threne	Anthra- cene	Fluoran- thene
<b>BIN C</b>									
379GLBR-325	19NOV4:504SC20	17	9 U	16 U	12 U	18	147	24	345
379GLBR-337	20NOV8:157SC20	45	13	19	48	82	517	98	1205
379GLBR-340	20NOV11:443SC30	64	39	56	88	59	553	112	1076
379GLBR-342	20NOV1:003SC30	53	37	69	95	74	761	175	1585
379GLBR-344	20NOV1:553SC30	49	27	44	71	50	530	110	1102
379GLBR-350	20NOV3:154SC30	36	12	13	27	32	268	42	599
379GLBR-351	20NOV3:154SC30	19	14 U	26 U	20 U	24	235	56	622
379GLBR-352	20NOV3:154SC30	19	15	14 U	21	32	280	69	735
379GLBR-357	20NOV3:337SC30	326	29	69 #	265	1219	2295 D	290	2195 D
379GLBR-360	20NOV3:278SC30	36	7 U	12 U	19	43	211	41	416
<b>BIN D</b>									
379GLBR-19	07OCT2:041SD0	98	41	49	78	49	546	120	1352
379GLBR-24	07OCT2:121SD0	97	36	43	71	42	499	106	1360
379GLBR-46	08OCT5:132SD30	1134	498	3656	4312 D	1700	33186 D	3081 D	39360 D
379GLBR-46-R	08OCT5:132SD30	58	53	56	86	52	655	144	1242
379GLBR-51	08OCT5:402SD10	157	40	697	646	397	6699 D	1358	10290 D
379GLBR-53	08OCT5:302SD20	199	57	616	599	383	5987 D	1261	9639 D
379GLBR-376, Rep 1	21NOV12:003SD10	61	34	71	104	71	703	173	1426
379GLBR-376, Rep 2	21NOV12:003SD10	33	31	46	70	55	520	130	1067
379GLBR-376, Rep 3	21NOV12:003SD10	68	31	54	79	55	522	132	1117
379GLBR-378	21NOV1:353SD10	237	45	510	454	302	4506 D	360	5568 D
379GLBR-380	21NOV2:433SD10	62	27	42	59	39	402	103	892
379GLBR-384	21NOV5:078SD10	108	9 U	15 U	37	51	299	57	709
379GLBR-388	21NOV5:107SD10	103	24	41 #	168	276	1455	267	3825 D
379GLBR-388-R	21NOV5:107SD10	187	46 U	81 U	173	289	1785	483	4220
379GLBR-395	21NOV5:224SD10	40	5 U	8 U	7 U	10	54	8	104
379GLBR-396	21NOV5:224SD10	14	6 U	10 U	9 U	5 U	34	5 U	76
379GLBR-397	21NOV5:224SD10	16	7 U	13 U	11 U	7 U	29	7 U	61

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>BIN C</b>								
379GLBR-325	19NOV4:504SC2O	208	148	250	302	196	112	160
379GLBR-337	20NOV8:157SC2O	886	601	950	1446 #	773 #	682	600
379GLBR-340	20NOV11:443SC3O	851	398	555	616	449	511	335
379GLBR-342	20NOV1:003SC3O	1156	599	728	828	614	596	465
379GLBR-344	20NOV1:553SC3O	808	396	530	618	457	404	357
379GLBR-350	20NOV3:154SC3O	434	223	378	457	289	255	208
379GLBR-351	20NOV3:154SC3O	448	276	386	476	316	279	261
379GLBR-352	20NOV3:154SC3O	528	330	449	554	389	368	314
379GLBR-357	20NOV3:337SC3O	2972 D	2064 D	4345 D	3580 D	5 U	2510 D	886
379GLBR-360	20NOV3:278SC3O	315	235	382	533	281	274	243
<b>BIN D</b>								
379GLBR-19	07OCT2:041SDO	980	490	672	815	515	592	451
379GLBR-24	07OCT2:121SDO	1000	494	696	830	527	587	444
379GLBR-46	08OCT5:132SD3O	25732 D	13327 D	14210 D	13802 D	11813 D	5 U	7382 D
379GLBR-46-R	08OCT5:132SD3O	946	444	620	621	486	478	364
379GLBR-51	08OCT5:402SD1O	7635 D	3449 D	3480 D	5527 D	2849	3303	2363
379GLBR-53	08OCT5:302SD2O	7165 D	3517	3367	3646	2862	3564	2233
379GLBR-376, Rep 1	21NOV12:003SD1O	1078	543	656	805	533	632	451
379GLBR-376, Rep 2	21NOV12:003SD1O	824	408	543	652	448	485	376
379GLBR-376, Rep 3	21NOV12:003SD1O	860	422	548	657	443	475	374
379GLBR-378	21NOV1:353SD1O	4717 D	2147 D	2736 D	2658 D	1901 D	2778 D	1395
379GLBR-380	21NOV2:433SD1O	673	335	440	528	351	344	292
379GLBR-384	21NOV5:078SD1O	532	481	784	1514	821	504	559
379GLBR-388	21NOV5:107SD1O	2867 D	1597 D	2794 D	6742	6 U	1418 #	1386
379GLBR-388-R	21NOV5:107SD1O	3297	2155	3345	5641	17 U	1616	1147
379GLBR-395	21NOV5:224SD1O	75	52	100	233	9	41	44
379GLBR-396	21NOV5:224SD1O	48	31	56	60	32	7	14
379GLBR-397	21NOV5:224SD1O	34	23	46	85	3 U	4 U	12

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	% Surrogate Recovery			
				d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo[a,h]Anthracene

<b>BIN C</b>							
379GLBR-325	19NOV4:504SC20	69	160	49%	40%	60%	71%
379GLBR-337	20NOV8:157SC20	245	631	74%	71%	88%	105%
379GLBR-340	20NOV11:443SC30	116	349	74%	70%	90%	100%
379GLBR-342	20NOV1:003SC30	169	447	57%	54%	89%	114%
379GLBR-344	20NOV1:553SC30	99	342	49%	42%	74%	94%
379GLBR-350	20NOV3:154SC30	79	225	77%	73%	95%	109%
379GLBR-351	20NOV3:154SC30	95	251	50%	46%	72%	85%
379GLBR-352	20NOV3:154SC30	115	307	38% *	38% *	81%	97%
379GLBR-357	20NOV3:337SC30	661	1960	72%	71%	92%	131% *
379GLBR-360	20NOV3:278SC30	95	269	64%	65%	83%	95%

<b>BIN D</b>							
379GLBR-19	07OCT2:041SD0	178	412	81%	67%	95%	131% *
379GLBR-24	07OCT2:121SD0	165	398	90%	72%	104%	133% *
379GLBR-46	08OCT5:132SD30	2276 D	6885 D	87%	65%	99%	114%
379GLBR-46-R	08OCT5:132SD30	92	373	73%	97%	89%	99%
379GLBR-51	08OCT5:402SD10	978	1997	38% *	32% *	50%	66%
379GLBR-53	08OCT5:302SD20	892	2029	93%	76%	117%	162% *
379GLBR-376, Rep 1	21NOV12:003SD10	164	434	50%	54%	94%	112%
379GLBR-376, Rep 2	21NOV12:003SD10	136	363	18% *	42%	77%	106%
379GLBR-376, Rep 3	21NOV12:003SD10	134	361	48%	42%	80%	95%
379GLBR-378	21NOV1:353SD10	581	1568	78%	72%	92%	104%
379GLBR-380	21NOV2:433SD10	79	281	55%	43%	86%	105%
379GLBR-384	21NOV5:078SD10	237	603	75%	71%	88%	128% *
379GLBR-388	21NOV5:107SD10	593	1538	66%	72%	93%	161% *
379GLBR-388-R	21NOV5:107SD10	521	1273	72%	101%	85%	103%
379GLBR-395	21NOV5:224SD10	23	44	69%	67%	84%	89%
379GLBR-396	21NOV5:224SD10	8	11	52%	32% *	73%	40%
379GLBR-397	21NOV5:224SD10	7	8	50%	27% *	65%	41%



**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Dibenzothiophene	Phenanthrene	Anthracene	Fluoranthene
Blank-1		57	7 U	12 U	9 U	7 U	6	6 U	8
Blank-2		22	20 U	34 U	27 U	18 U	15 U	17 U	12 U
Blank-3		14 U	11 U	20 U	16 U	10 U	8 U	10 U	7 U
Blank-4		16	12 U	22 U	20 U	13 U	11 U	13 U	11 U
Blank-5		46	8 U	13 U	11 U	8 U	7	8 U	8
Blank-R		16	7 U	13 U	9 U	7 U	6	6 U	5 U
Blank-Diluted		91 U	80 U	134 U	99 U	70 U	55 U	70 U	53 U

**STANDARD REFERENCE MATERIAL**

SRM-1 (HS-2) (1)	250	157	97	209	221	2750	278	6622
SRM-2 (HS-2)	138	148	87	175	188	2495	269	5588
SRM-3 (HS-2)	92	136	62	177	179	2411 E	243	5811 E
SRM-4 (HS-2)	154	123	68	174	172	2181	212	5451 E
SRM-5 (HS-2)	201	151	78	225	225	2916 E	307	6482 E
	36%	10%	18%	12%	12%	11%	14%	9%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]-anthracene	Chrysene	Benzo(b)-Fluoranthene	Benzo(k)-Fluoranthene	Benzo[a]-pyrene	Indeno [1,2,3-cd]-pyrene
Blank-1		5 U	5 U	5 U	5 U	4 U	5 U	3 U
Blank-2		11 U	10 U	9 U	9 U	8 U	9 U	6 U
Blank-3		6 U	6 U	5 U	6 #	4 U	6 U	4 U
Blank-4		10 U	10 U	9 U	9 U	8 U	10 U	6 U
Blank-5		9	5	11	13	7	5 U	5
Blank-R		5 U	4 U	4 U	5	3 U	4 U	3 U
Blank-Diluted		48 U	45 U	42 U	42 U	36 U	45 U	26 U

**STANDARD REFERENCE MATERIAL**

SRM-1 (HS-2) (1)	4121	1800	2170	2024	1415	793	867
SRM-2 (HS-2)	3513	1568	1930	1765	1510	971	779
SRM-3 (HS-2)	3501 E	1642	1919 E	2118 E	1506	973	894
SRM-4 (HS-2)	3375 E	1447	1704 E	1745	1404	986	759
SRM-5 (HS-2)	4127 E	1770 E	2217 E	2487 E	1798	1159	925
	10%	9%	11%	15%	10%	13%	9%
RSD%							
NIST 1941, Rep 1	938	435	590	825	611	457	453
NIST 1941, Rep 2	940	450	612	842	603	460	440
NIST 1941, Rep 3	1024	488	663	935	638	499	487
certified value	1080	550	NA	780	444	670	569
	±200	±79	NA	±190	±49	±130	±40

MSL Code	Sponsor ID	Dibenzo[a,h]-		Benzo[ghi] perylene	% Surrogate Recovery					
		anthracene	pyrene		d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Anthracene (a,h)		
Blank-1		4 U		4 U	71%		1% *		84%	16% *
Blank-2		7 U		6 U	63%		31% *		71%	62%
Blank-3		4 U		4 U	1% *		14% *		46%	62%
Blank-4		7 U		7 U	51%		12% *		71%	50%
Blank-5		4 U		5	71%		67%		85%	96%
Blank-R		3 U		3 U	72%		81%		79%	76%
Blank-Diluted		32 U		28 U	0% *		14% *		40%	48%

[illegible]

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

(Concentrations in ng/g)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Flourene	Dibenzothiophene	Phenanthrene	Anthracene	Fluoranthene
<b>SPIKE RESULTS</b>									
Amount Spiked		167	167	167	167	167	167	167	167
Blank-4		16	12 U	22 U	20 U	13 U	11 U	13 U	11 U
Blank-4 + Spike		120	53	103	133	81	137	44	187 E
Amount Recovered		104	53	103	133	81	137	44	187 E
Percent Recovery		62%	32% *	62%	80%	49%	82%	26% *	112%
Amount Spiked		167	167	167	167	167	167	167	167
Blank-5		46	8 U	13 U	11 U	8 U	7	8 U	8
Blank-5+ Spike		168	139	151	161	146	159	107	186
Amount Recovered		123	139	151	161	146	152	107	178
Percent Recovery		73%	83%	90%	96%	87%	91%	64%	107%
Amount Spiked		167	167	167	167	167	167	167	167
Blank-5		46	8 U	13 U	11 U	8 U	7	8 U	8
Blank-5+ Spike		88	107	118	128	118	130	93	158
Amount Recovered		42	107	118	128	118	123	93	150
Percent Recovery		25% *	64%	71%	77%	71%	74%	56%	90%
Amount Spiked		167	167	167	167	167	167	167	167
Blank-R		16	7 U	13 U	9 U	7 U	6	6 U	5 U
Blank-R + Spike		138	124	141	135	152	166	153	171
Amount Recovered		122	124	141	135	152	160	153	171
Percent Recovery		73%	74%	84%	81%	91%	96%	92%	103%
Amount Spiked		167	167	167	167	167	167	167	167
Blank-R		16	7 U	13 U	9 U	7 U	6	6 U	5 U
Blank-R + Spike		137	135	152	148	158	169	145	176
Amount Recovered		121	135	152	148	158	163	145	176
Percent Recovery		72%	81%	91%	89%	95%	98%	87%	105%

DUPLICATE

DUPLICATE

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
<b>SPIKE RESULTS</b>								
Amount Spiked		167	167	167	167	167	167	167
Blank-4		10 U	10 U	9 U	9 U	8 U	10 U	6 U
Blank-4 + Spike		148 E	130	147	195	186	11 U	121
Amount Recovered		148 E	130	147	195	186	11 U	121
Percent Recovery		102%	78%	88%	117%	111%	NA	72%
Amount Spiked		167	167	167	167	167	167	167
Blank-5		9	5	11	13	7	5 U	5
Blank-5+ Spike		168	150	155	220	210	136	117
Amount Recovered		160	145	144	207	203	136	112
Percent Recovery		96%	87%	86%	124% *	121% *	82%	67%
Amount Spiked		167	167	167	167	167	167	167
Blank-5		9	5	11	13	7	5 U	5
Blank-5+ Spike		144	134	136	187	189	122	127
Amount Recovered		135	129	125	175	181	122	123
Percent Recovery		81%	77%	75%	105%	109%	73%	73%
Amount Spiked		167	167	167	167	167	167	167
Blank-R		5 U	4 U	4 U	5	3 U	4 U	3 U
Blank-R + Spike		168	157	159	167	167	140	164
Amount Recovered		168	157	159	162	167	140	164
Percent Recovery		100%	94%	95%	97%	100%	84%	98%
Amount Spiked		167	167	167	167	167	167	167
Blank-R		5 U	4 U	4 U	5	3 U	4 U	3 U
Blank-R + Spike		167	158	161	186	187	139	178
Amount Recovered		167	158	161	167	187	139	178
Percent Recovery		100%	95%	96%	100%	112%	83%	107%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene		Benzo[ghi]perylene	% Surrogate Recovery			
		167	7 U		d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo[a,h]Anthracene

**SPIKE RESULTS**

Amount Spiked		167		167	NA	NA	NA	NA
Blank-4		7 U		7 U	51%	12% *	71%	50%
Blank-4 + Spike		173		91	49%	42%	66%	78%
Amount Recovered		173		91	NA	NA	NA	NA
Percent Recovery		104%		55%	NA	NA	NA	NA
Amount Spiked		167		167	NA	NA	NA	NA
Blank-5		4 U		5	71%	67%	85%	96%
Blank-5+ Spike		170		117	62%	61%	78%	88%
Amount Recovered		170		112	NA	NA	NA	NA
Percent Recovery		102%		67%	NA	NA	NA	NA
Amount Spiked		167		167	NA	NA	NA	NA
Blank-5		4 U		5	71%	67%	85%	96%
Blank-5+ Spike		142		97	31% *	57%	75%	85%
Amount Recovered		142		92	NA	NA	NA	NA
Percent Recovery		85%		55%	NA	NA	NA	NA
Amount Spiked		167		167	NA	NA	NA	NA
Blank-R		3 U		3 U	72%	81%	79%	76%
Blank-R + Spike		145		127	71%	82%	83%	84%
Amount Recovered		145		127	NA	NA	NA	NA
Percent Recovery		87%		76%	NA	NA	NA	NA
Amount Spiked		167		167	NA	NA	NA	NA
Blank-R		3 U		3 U	72%	81%	79%	76%
Blank-R + Spike		160		138	70%	83%	85%	88%
Amount Recovered		160		138	NA	NA	NA	NA
Percent Recovery		96%		83%	NA	NA	NA	NA

DUPLICATE

DUPLICATE

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Dibenzothiophene	Phenanthrene	Anthracene	Fluoranthene
<b>SPIKE RESULTS</b>									
Amount Spiked									
379GLBR-111-R	22OCT5:563SA20	303	303		303	303	303	303	303
379GLBR-111-R + Spike		36 U	44	53 U	46	32	446	93	978
Amount Recovered		272	277	314	310	326	767	382	1333
Percent Recovery		262	233	314	264	294	321	289	355
		87%	77%	104%	87%	97%	106%	96%	117%
Amount Spiked									
379GLBR-111-R	22OCT5:563SA20	312	312	312	312	312	312	312	312
379GLBR-111-R + Spike	22OCT5:563SA20	36 U	44	53 U	46	32	446	93	978
Amount Recovered	DUPLICATE	94	314	396	305	347	1128	426	2111
Percent Recovery		94	270	396	260	315	683	333	1132
		30% *	87%	127% *	83%	101%	219% *	107%	363% *
Amount Spiked									
379GLBR-247	31OCT2:384SB30	98	98	98	98	98	98	98	98
379GLBR-247 + Spike		21	12 U	22 #	14 U	16	90	13	166
Amount Recovered		73	47	189 #	69	76	157	54	255
Percent Recovery		52	47	167	69	60	67	41	89
		53%	48%	170% *	70%	61%	68%	42%	91%
Amount Spiked									
379GLBR-247	31OCT2:384SB30	100	100	100	100	100	100	100	100
379GLBR-247 + Spike		21	12 U	22 #	14 U	16	90	13	166
Amount Recovered	DUPLICATE	67	40	166	63	76	139	52	210
Percent Recovery		46	40	144	63	60	49	39	44
		46%	40%	145% *	63%	60%	49%	39% *	44%
Amount Spiked									
379GLBR-247-R	31OCT2:384SB30	154	154	154	154	154	154	154	154
379GLBR-247-R + Spike		45 U	39 U	68 U	9 U	21	141	18	171
Amount Recovered		139	93	131	127	147	261	127	359
Percent Recovery		139	93	131	127	126	119	109	187
		90%	60%	85%	82%	82%	77%	71%	122% *

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

(Concentrations in ng/g)

MSL Code	Sponsor ID	Pyrene	Benzo[a]-anthracene	Chrysene	Benzo(b)-Fluoranthene	Benzo(k)-Fluoranthene	Benzo[a]-pyrene	Indeno [1,2,3-cd]-pyrene
<b>SPIKE RESULTS</b>								
Amount Spiked		303	303	303	303	303	303	303
379GLBR-111-R	22OCT5:563SA20	737	339	487	507	373	372	294
379GLBR-111-R + Spike		1069	643	795	849	664	643	632
Amount Recovered		332	304	308	342	290	270	339
Percent Recovery		109%	100%	102%	113%	96%	89%	112%
Amount Spiked		312	312	312	312	312	312	312
379GLBR-111-R	22OCT5:563SA20	737	339	487	507	373	372	294
379GLBR-111-R + Spike	DUPLICATE	1654	1009	1277	1264	1031	977	844
Amount Recovered		916	671	790	757	657	604	550
Percent Recovery		294%*	215%*	253%*	243%*	211%*	194%*	176%*
Amount Spiked		98	98	98	98	98	98	98
379GLBR-247	31OCT2:384SB30	109	73	132	253	4 U	53	67
379GLBR-247 + Spike		177	142	207	225	171	50	118
Amount Recovered		68	69	75	-28	171	-3	51
Percent Recovery		69%	70%	77%	-29%*	174%*	-3%*	52%
Amount Spiked		100	100	100	100	100	100	100
379GLBR-247	31OCT2:384SB30	109	73	132	253	4 U	53	67
379GLBR-247 + Spike	DUPLICATE	152	123	174	184	141	32	83
Amount Recovered		43	50	42	-69	141	-21	16
Percent Recovery		43%	50%	42%	-69%*	142%*	-21%*	16%*
Amount Spiked		154	154	154	154	154	154	154
379GLBR-247-R	31OCT2:384SB30	121	87	156	262	3 U	60	59
379GLBR-247-R + Spike		278	215	282	309	237	154	165
Amount Recovered		157	129	126	47	234	94	107
Percent Recovery		102%	84%	82%	31%*	152%*	61%	69%



**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

MSL Code		Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	% Surrogate Recovery			
					d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo[a,h]Anthracene
SPIKE RESULTS								
Amount Spiked			303	303	NA	NA	NA	NA
379GLBR-111-R		22OCT5:563SA20	74	294	33% *	72%	65%	91%
379GLBR-111-R + Spike			404	579	67%	85%	83%	95%
Amount Recovered			330	285	NA	NA	NA	NA
Percent Recovery			109%	94%	NA	NA	NA	NA
Amount Spiked			312	312	NA	NA	NA	NA
379GLBR-111-R		22OCT5:563SA20	74	294	33% *	72%	65%	91%
379GLBR-111-R + Spike		DUPLICATE	506	759	21% *	95%	75%	95%
Amount Recovered			432	465	NA	NA	NA	NA
Percent Recovery			139% *	149% *	NA	NA	NA	NA
Amount Spiked			98	98	NA	NA	NA	NA
379GLBR-247		31OCT2:384SB30	32	64	50%	39% *	55%	63%
379GLBR-247 + Spike			97	92	50%	45%	58%	63%
Amount Recovered			65	28	NA	NA	NA	NA
Percent Recovery			66%	29% *	NA	NA	NA	NA
Amount Spiked			100	100	NA	NA	NA	NA
379GLBR-247		31OCT2:384SB30	32	64	50%	39% *	55%	63%
379GLBR-247 + Spike		DUPLICATE	78	61	43%	40%	53%	55%
Amount Recovered			46	-3	NA	NA	NA	NA
Percent Recovery			46%	-3% *	NA	NA	NA	NA
Amount Spiked			154	154	NA	NA	NA	NA
379GLBR-247-R		31OCT2:384SB30	30	62	1% *	131% *	52%	67%
379GLBR-247-R + Spike			136	142	67%	75%	74%	63%
Amount Recovered			106	80	NA	NA	NA	NA
Percent Recovery			69%	52%	NA	NA	NA	NA

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Dibenzothiophene	Phenanthrene	Anthracene	Fluoranthene
<b>SPIKE RESULTS</b>									
Amount Spiked									
379GLBR-247-R	31OCT2:384SB30	142	142	142	142	142	142	142	142
379GLBR-247-R + Spike	DUPLICATE	45 U	39 U	68 U	9 U	21	141	18	171
Amount Recovered		34 U	97	193	79	151	282	129	341
Percent Recovery		NA	97	193	79	130	141	111	170
		NA	69%	136%	55%	92%	99%	78%	120%
Amount Spiked									
379GLBR-268	1NOV10:208SB30	109	109	109	109	109	109	109	109
379GLBR-268 + Spike		81	8 U	15 U	34	74	364	60	763
Amount Recovered		133	61	82	112	142	390	101	757
Percent Recovery		52	61	82	78	68	26	41	-6
		48%	56%	76%	71%	62%	24%	38%	-6%
Amount Spiked									
379GLBR-268	1NOV10:208SB30	96	96	96	96	96	96	96	96
379GLBR-268 + Spike	DUPLICATE	81	8 U	15 U	34	74	364	60	763
Amount Recovered		141	56	80	123	154	444	111	865
Percent Recovery		59	56	80	88	80	80	51	102
		62%	58%	83%	92%	83%	84%	53%	106%
Amount Spiked									
379GLBR-388	21NOV5:107SD10	107	107	107	107	107	107	107	107
379GLBR-388 + Spike		103	24	41 #	168	276	1455	267	3825 D
Amount Recovered		186	112	140	281	352	1501 D	311	3923 D
Percent Recovery		83	88	99	113	76	46	45	98
		78%	82%	93%	105%	71%	43%	42%	92%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]-anthracene	Chrysene	Benzo(b)-Fluoranthene	Benzo(k)-Fluoranthene	Benzo[a]-pyrene	Indeno [1,2,3-cd]-pyrene
<b>SPIKE RESULTS</b>								
Amount Spiked		142	142	142	142	142	142	142
379GLBR-247-R	31OCT2:384SB30	121	87	156	262	3 U	60	59
379GLBR-247-R + Spike	DUPLICATE	270	213	289	303	229	149	163
Amount Recovered		149	127	132	41	227	89	105
Percent Recovery		105%	89%	93%	29%*	160%*	62%	74%
Amount Spiked		109	109	109	109	109	109	109
379GLBR-268	1NOV10:208SB30	475	360	562	806	422 #	281	249
379GLBR-268 + Spike		495	395	563	760	479	264	300
Amount Recovered		20	35	1	-47	57	-18	51
Percent Recovery		18%*	32%*	1%*	-43%*	52%	-16%*	47%
Amount Spiked		96	96	96	96	96	96	96
379GLBR-268	1NOV10:208SB30	475	360	562	806	422 #	281	249
379GLBR-268 + Spike	DUPLICATE	559	435	637	878	506	282	323
Amount Recovered		84	75	75	72	84	1	74
Percent Recovery		88%	78%	78%	75%	88%	1%*	77%
Amount Spiked		107	107	107	107	107	107	107
379GLBR-388	21NOV5:107SD10	2867 D	1597 D	2794 D	6742	6 U	1418 #	1386
379GLBR-388 + Spike		2954 D	1695 D	2921 D	7409	5 U	1572 #	1655
Amount Recovered		87	98	127	667	5	154	269
Percent Recovery		81%	92%	119%	623%*	NA	144%*	251%*

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

				% Surrogate Recovery			
MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo-(a,h)Anthracene
SPIKE RESULTS							
Amount Spiked		142	142	NA	NA	NA	NA
379GLBR-247-R	31OCT2:384SB30	30	62	1% *	131% *	52%	67%
379GLBR-247-R + Spike	DUPLICATE	130	139	1% *	118%	51%	70%
Amount Recovered		100	77	NA	NA	NA	NA
Percent Recovery		70%	54%	NA	NA	NA	NA
Amount Spiked		109	109	NA	NA	NA	NA
379GLBR-268	1NOV10:208SB30	108	224	61%	31% *	71%	64%
379GLBR-268 + Spike		209	243	56%	52%	68%	76%
Amount Recovered		101	19	NA	NA	NA	NA
Percent Recovery		92%	18% *	NA	NA	NA	NA
Amount Spiked		96	96	NA	NA	NA	NA
379GLBR-268	1NOV10:208SB30	108	224	61%	31% *	71%	64%
379GLBR-268 + Spike	DUPLICATE	222	267	63%	56%	82%	85%
Amount Recovered		114	44	NA	NA	NA	NA
Percent Recovery		119%	45%	NA	NA	NA	NA
Amount Spiked		107	107	NA	NA	NA	NA
379GLBR-388	21NOV5:107SD10	593	1538	66%	72%	93%	161%
379GLBR-388 + Spike		943	1778	71%	72%	92%	167%
Amount Recovered		350	240	NA	NA	NA	NA
Percent Recovery		327% *	224% *	NA	NA	NA	NA

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naph- thalene	Acenaph- thylene	Acenaph- thene	Flourene	Dibenzo- thiophene	Phenan- threne	Anthra- cene	Fluoran- thene
<b>SPIKE RESULTS</b>									
Amount Spiked		117	117	117	117	117	117	117	117
379GLBR-388	21NOV5:107SD10	103	24	41 #	168	276	1455	267	3825 D
379GLBR-388 + Spike	DUPLICATE	194	126	160	296	370	1503 D	316	3853 D
Amount Recovered		91	101	118	128	95	48	49	28
Percent Recovery		77%	87%	101%	110%	81%	41%	42%	24% *
Amount Spiked		500	500	500	500	500	500	500	500
379GLBR-388-R	21NOV5:107SD10	187	46 U	81 U	173	289	1785	483	4220
379GLBR-388-R + Spike		549	470	547	661	800	2263	915	4951
Amount Recovered		362	470	547	488	511	478	432	732
Percent Recovery		72%	94%	109%	98%	102%	96%	86%	146% *
Amount Spiked		385	385	385	385	385	385	385	385
379GLBR-388-R	21NOV5:107SD10	187	46 U	81 U	173	289	1785	483	4220
379GLBR-388-R + Spike	DUPLICATE	378	325	390	511	664	2080	777	4828
Amount Recovered		191	325	390	338	375	295	294	609
Percent Recovery		50%	84%	101%	88%	97%	77%	76%	158% *
<b>REPLICATE ANALYSIS</b>									
379GLBR-182, Rep 1	25OCT2:294SB10	27	7 U	12 U	16	11	146	18	274
379GLBR-182, Rep 2	25OCT2:294SB10	22	11 U	69 #	21	18	191	35	356
379GLBR-182, Rep 3	25OCT2:294SB10	26	11 U	72 #	16	14	143	29	277
	RSD %	11%	NA	4%	16%	25% *	17%	32% *	15%
379GLBR-199, Rep 1	20OCT3:573SB20	62	31	52	74	48	503	91	1123
379GLBR-199, Rep 2	20OCT3:573SB20	49	29	42	59	40	424	81	931
379GLBR-199, Rep 3	20OCT3:573SB20	59	23	40	61	44	472	93	892
	RSD %	12%	15%	14%	13%	10%	9%	7%	13%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]- anthracene	Chrysene	Benzo(b)- Fluoranthene	Benzo(k)- Fluoranthene	Benzo[a]- pyrene	Indeno [1,2,3-cd]- pyrene
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**SPIKE RESULTS**

Amount Spiked		117	117	117	117	117	117	117
379GLBR-388	21NOV5:107SD10	2867 D	1597 D	2794 D	6742	6 U	1418 #	1386
379GLBR-388 + Spike	DUPLICATE	2927 D	1682 D	2925 D	7141 E	5 U	1545 #	1573
Amount Recovered		60	85	131	399	5	127	187
Percent Recovery		51%	73%	112%	341% *	NA	108%	160% *
Amount Spiked		500	500	500	500	500	500	500
379GLBR-388-R	21NOV5:107SD10	3297	2155	3345	5641	17 U	1616	1147
379GLBR-388-R + Spike		3889	2583	3543	3969 #	2271 #	1965	1701
Amount Recovered		592	428	198	-1672	2271 #	348	555
Percent Recovery		118%	86%	40%	-334% *	454% *	70%	111%
Amount Spiked		385	385	385	385	385	385	385
379GLBR-388-R	21NOV5:107SD10	3297	2155	3345	5641	17 U	1616	1147
379GLBR-388-R + Spike	DUPLICATE	3805	2484	3547	3889 #	2005 #	1876	1563
Amount Recovered		508	329	201	-1752	2005	260	416
Percent Recovery		132% *	85%	52%	-455% *	521% *	67%	108%

**REPLICATE ANALYSIS**

379GLBR-182, Rep 1	25OCT2:294SB10	173	98	187	243	147	29	104
379GLBR-182, Rep 2	25OCT2:294SB10	254	145	217	239	154	97	107
379GLBR-182, Rep 3	25OCT2:294SB10	196	122	195	231	137	94	100
	RSD %	20%	19%	8%	3%	6%	52% *	3%
379GLBR-199, Rep 1	20OCT3:573SB20	833	376	517	631	457	447	355
379GLBR-199, Rep 2	20OCT3:573SB20	696	322	433	499	378	383	295
379GLBR-199, Rep 3	20OCT3:573SB20	649	298	410	470	335	343	268
	RSD %	13%	12%	12%	16%	16%	13%	15%

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

		Dibenzo[a,h]-anthracene		Benzo[ghi]perylene	% Surrogate Recovery			
MSL Code	Sponsor ID				d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo-(a,h)Anthracene
SPIKE RESULTS								
Amount Spiked		117		117	NA	NA	NA	NA
379GLBR-388	21NOV5:107SD10	593		1538	66%	72%	93%	161%
379GLBR-388 + Spike	DUPLICATE	914		1700	63%	69%	87%	154%
Amount Recovered		322		162	NA	NA	NA	NA
Percent Recovery		275% *		138% *	NA	NA	NA	NA
Amount Spiked		500		500	NA	NA	NA	NA
379GLBR-388-R	21NOV5:107SD10	521		1273	72%	101%	85%	103%
379GLBR-388-R + Spike		1067		1748	77%	91%	92%	109%
Amount Recovered		546		476	NA	NA	NA	NA
Percent Recovery		109%		95%	NA	NA	NA	NA
Amount Spiked		385		385	NA	NA	NA	NA
379GLBR-388-R	21NOV5:107SD10	521		1273	72%	101%	85%	103%
379GLBR-388-R + Spike	DUPLICATE	952		1657	63%	81%	83%	108%
Amount Recovered		431		384	NA	NA	NA	NA
Percent Recovery		112%		100%	NA	NA	NA	NA
REPLICATE ANALYSIS								
379GLBR-182, Rep 1	25OCT2:294SB10	48		90	62%	45%	78%	89%
379GLBR-182, Rep 2	25OCT2:294SB10	51		99	47%	43%	64%	73%
379GLBR-182, Rep 3	25OCT2:294SB10	48		96	66%	62%	78%	87%
	RSD %	4%		5%	NA	NA	NA	NA
379GLBR-199, Rep 1	20OCT3:573SB20	130		353	87%	73%	94%	105%
379GLBR-199, Rep 2	20OCT3:573SB20	106		291	74%	62%	78%	87%
379GLBR-199, Rep 3	20OCT3:573SB20	96		253	70%	61%	78%	89%
	RSD %	16%		17%	NA	NA	NA	NA

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Dibenzothiophene	Phenanthrene	Anthracene	Fluoranthene
<b>REPLICATE ANALYSIS</b>									
379GLBR-18, Rep 1	07OCT1:331SCO	147	66	92	147	105	923	264	1938
379GLBR-18, Rep 2	07OCT1:331SCO	94	41	67	95	75	615	167	1343
379GLBR-18, Rep 3	07OCT1:331SCO	125	53	91	131	100	835	237	1793
	<b>RSD %</b>	<b>22% *</b>	<b>23% *</b>	<b>17%</b>	<b>21% *</b>	<b>17%</b>	<b>20%</b>	<b>23% *</b>	<b>18%</b>
379GLBR-323, Rep 1	19NOV4:504SC20	36	7 U	12 U	10 U	24	128	20	267
379GLBR-323, Rep 2	19NOV4:504SC20	31	10	10 U	13	28	174	28	379
379GLBR-323, Rep 3	19NOV4:504SC20	31	7 U	11 U	9 U	22	125	20	265
	<b>RSD %</b>	<b>9%</b>	<b>NA</b>	<b>NA</b>	<b>NA</b>	<b>12%</b>	<b>19%</b>	<b>21% *</b>	<b>22% *</b>
379GLBR-376, Rep 1	21NOV12:003SD10	61	34	71	104	71	703	173	1426
379GLBR-376, Rep 2	21NOV12:003SD10	33	31	46	70	55	520	130	1067
379GLBR-376, Rep 3	21NOV12:003SD10	68	31	54	79	55	522	132	1117
	<b>RSD %</b>	<b>34% *</b>	<b>6%</b>	<b>22% *</b>	<b>21% *</b>	<b>15%</b>	<b>18%</b>	<b>17%</b>	<b>16%</b>

\* - Recoveries outside of QC limits.

U - Detected at or below detection limit.

R - Rerun samples.

D - Sample diluted 1:10.

E - Value out of calibration range.

# - Ion ratio out of specification.

(1) - HS-2 is not certified for PAHs. However, RSD values give an indication of relative precision of PAH measurements between batches.

NA - Not applicable/analyzed.

RSD% - Relative Standard Deviation



**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

*(Concentrations in ng/g)*

MSL Code	Sponsor ID	Pyrene	Benzo[a]-anthracene	Chrysene	Benzo(b)-Fluoranthene	Benzo(k)-Fluoranthene	Benzo[a]-pyrene	Indeno[1,2,3-cd]-pyrene
379GLBR-18, Rep 1	07OCT1:331SCO	1554	785	957	1039	777	912	621
379GLBR-18, Rep 2	07OCT1:331SCO	1077	528	664	766	513	601	438
379GLBR-18, Rep 3	07OCT1:331SCO	1436	747	893	1033	695	852	583
	RSD %	18%	20%	18%	16%	20%	21%	18%
379GLBR-323, Rep 1	19NOV4:504SC20	189	131	238	484	4 U	131	137
379GLBR-323, Rep 2	19NOV4:504SC20	277	178	296	361	229	189	180
379GLBR-323, Rep 3	19NOV4:504SC20	180	126	240	308	174	123	141
	RSD %	25%	20%	13%	24%	27%	24%	16%
379GLBR-376, Rep 1	21NOV12:003SD10	1078	543	656	805	533	632	451
379GLBR-376, Rep 2	21NOV12:003SD10	824	408	543	652	448	485	376
379GLBR-376, Rep 3	21NOV12:003SD10	860	422	548	657	443	475	374
	RSD %	15%	16%	11%	12%	11%	17%	11%

\* = Recoveries outside of QC limits.

U = Detected at or below detection limit.

R = Rerun samples.

D = Sample diluted 1:10.

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# = Ion ratio out of specification.

(1) = HS-2 is not certified for PAHs. However, RSD values give an indication of relative precision of PAH measurements between batches.

NA = Not applicable/analyzed.

RSD% = Relative Standard Deviation

**BUFFALO RIVER PILOT PROJECT (#379)  
PAH ANALYSIS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Dibenzo[a,h]-anthracene	Benzo[ghi]perylene	% Surrogate Recovery			
				d8 Naphthalene	d10 Acenaphthene	D10-Fluorene	D14 Dibenzo[a,h]Anthracene
REPLICATE ANALYSIS							
379GLBR-18, Rep 1	07OCT1:331SCO	251	602	64%	59%	89%	123%
379GLBR-18, Rep 2	07OCT1:331SCO	179	388	57%	61%	84%	121%
379GLBR-18, Rep 3	07OCT1:331SCO	241	505	72%	68%	96%	133%
	RSD %	18%	22%	NA	NA	NA	NA
379GLBR-323, Rep 1	19NOV4:504SC2O	54	144	86%	77%	89%	96%
379GLBR-323, Rep 2	19NOV4:504SC2O	71	189	84%	76%	91%	101%
379GLBR-323, Rep 3	19NOV4:504SC2O	55	146	83%	74%	88%	99%
	RSD %	15%	16%	NA	NA	NA	NA
379GLBR-376, Rep 1	21NOV12:003SD1O	164	434	50%	54%	94%	112%
379GLBR-376, Rep 2	21NOV12:003SD1O	136	363	18%	42%	77%	106%
379GLBR-376, Rep 3	21NOV12:003SD1O	134	361	38%	42%	80%	95%
	RSD %	11%	11%	NA	NA	NA	NA

\* = Recoveries outside of QC limits.

U = Detected at or below detection limit.

R = Rerun samples.

D = Sample diluted 1:10.

E = Value out of calibration range.

# = Ion ratio out of specification.

(1) = HS-2 is not certified for PAHs. However, RSD values give an indication of relative precision of PAH measurements between batches.

NA = Not applicable/analyzed.

RSD% = Relative Standard Deviation

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN WATER SAMPLES**

MSL Code	Dil.	Sponsor ID	Concentrations in ng/L				% Surrogate Recovery	
			Sample Amount (L)	Aroclor 1242/1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
BIN A								
379GLBR-97	(1:10)	21OCT6:1810LA10	0.810	21,111 D	500 U	500 U	36.1%	25.7%
379GLBR-97 ~		21OCT6:2010LA10	0.800	26,368	50 U	50 U	52.9%	38.6%
379GLBR-97 ~	(1:10)	21OCT6:2010LA10	0.800	22,742 D	500 U	500 U	113.4%	77.4%
379GLBR-128	(1:10)	23OCT4:459LA20	0.720	24,698 D(a)	500 U	500 U	103.1%	33.3%
379GLBR-132	(1:10)	23OCT5:2010LA20	0.675	60,890 D	5,294 E	500 U	53.0%	36.6%
379GLBR-160	(1:10)	25OCT10:1810LA30	0.700	21,020 D	500 U	500 U	113.6%	77.9%
379GLBR-161	(1:10)	25OCT10:1910LA30	0.760	33,666 D	500 U	500 U	135.7%	68.5%
379GLBR-164	(1:10)	25OCT9:549LA30	0.650	69,074 D	500 U	500 U	69.7%	59.2%
379GLBR-204		30OCT4:206LA30	0.800	200 U	50 U	50 U	76.1%	105.0%
BIN B								
379GLBR-187	(1:10)	25OCT3:0810LB10	0.750	27,605 D	500 U	500 U	103.0%	83.3%
379GLBR-226	(1:10)	31OCT10:129LB20	0.850	2,000 U	500 U	500 U	54.6%	33.1%
379GLBR-227	(1:10)	31OCT10:2310LB20	0.825	2,000 U	500 U	500 U	66.6%	45.4%
379GLBR-252	(1:10)	31OCT3:119LB30	0.825	2,000 U	500 U	500 U	113.5%	58.4%
379GLBR-253	(1:10)	31OCT3:109LB30	0.775	2,000 U	500 U	500 U	92.3%	50.0%
379GLBR-256	(1:10)	31OCT3:0110LB30	0.750	8,107 D	500 U	500 U	72.5%	55.5%
379GLBR-257	(1:10)	31OCT3:0010LB30	0.650	16,922 D	500 U	500 U	69.7%	47.9%
BIN C								
379GLBR-286	(1:10)	18NOV5:559LC10	0.775	25,956 D	500 U	500 U	116.9%	47.9%
379GLBR-326	(1:10)	19NOV5:509LC20	0.800	39,269 D	500 U	500 U	65.8%	44.5%
379GLBR-331	(1:10)	19NOV5:0510LC20	0.650	17,890 D	500 U	500 U	61.8%	54.9%
379GLBR-331 DUP 1	(1:10)	19NOV5:0510LC20	0.785	11,157 D	500 U	500 U	63.1%	58.3%
379GLBR-331 DUP 2	(1:10)	19NOV5:0510LC20	0.800	8,028 D	500 U	500 U	56.2%	53.8%
379GLBR-361		20NOV2:193LC30	0.800	200 U	50 U	50 U	38.8%	52.8%
379GLBR-366	(1:10)	20NOV2:419LC30	0.800	5,814 D	500 U	500 U	63.6%	50.3%
379GLBR-370	(1:10)	20NOV3:0110LC30	0.726	19,471 D	500 U	500 U	42.6%	51.8%

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN WATER SAMPLES**

MSL Code	Dil.	Sponsor ID	Concentrations in ng/L				% Surrogate Recovery	
			Sample Amount (L)	Aroclor 1242/1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Oclachloro-naphthalene

**BIN D**

379GLBR-398	(1:10)	21NOV3:2610LD10	0.757	5,304 D	500 U	500 U	45.7%	51.3%
379GLBR-404	(1:10)	21NOV4:169LD10	0.705	7,193 D	500 U	500 U	49.4%	27.3%

**DILUTION WATER**

379GLBR-275		6NOV12:003LO	0.825	200 U	50 U	50 U	36.8%	59.3%
379GLBR METHOD BLANK			0.500	200 U	50 U	50 U	62.4%	96.4%

**MATRIX SPIKE RESULTS**

Amount Spiked								
380GLBR-132	(1:10)	23OCT5:2010LA20	NS	NS	7,143	NS	NA	NA
380GLBR-132 + Spike			NS	NS	5,294 E	NS	53.0%	36.6%
Amount Recovered			NS	NS	13,287	NS	43.6%	49.5%
Percent Recovery			NS	NS	7,993	NS	NA	NA
			NS	NS	112%	NS	NA	NA

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN WATER SAMPLES**

MSL Code	Dil.	Sponsor ID	Sample Amount (L)	Concentrations in ng/L			% Surrogate Recovery	
				Aroclor 1242/1248	Aroclor 1254	Aroclor 1260	Tetrachloro- m-Xylene	Octachloro- naphthalene

**REPLICATE ANALYSIS**

379GLBR-331	(1:10)	19NOV5:0510LC20		17,890 D	500 U	500 U	61.8%	54.9%
379GLBR-331 DUP 1	(1:10)	19NOV5:0510LC20		11,157 D	500 U	500 U	63.1%	58.3%
379GLBR-331 DUP 2	(1:10)	19NOV5:0510LC20		8,028 D	500 U	500 U	56.2%	53.8%

RSD %

41% \*\*

NA

NA

NA

~ = Field replicate.

U = Not detected at detection limit shown.

D = Results from diluted sample extract.

(a) = Aroclor quantified with one peak; other peaks may have been present, but not quantifiable.

E = Estimated/most likely due to residual peaks of primary aroclor.

NS = Not spiked.

NA = Not applicable.

\* = Recoveries were outside laboratory control limits (40-120%).

\*\* = RSD exceeded QC limit of 20%.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	Concentrations In ng/g dry weight				% Surrogate Recovery	
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
BIN A									
379GLBR-58	09OCT11:382SA20	2/19/92	60.44	50 U	55 D	25 U	25 U	83.1%	136.8%
379GLBR-104	22OCT11:317SA10	2/19/92	65.53	50 U	50 U	25 U	25 U	65.1%	107.0%
379GLBR-107	22OCT11:378SA10	2/19/92	97.72	50 U	50 U	25 U	25 U	70.8%	108.2%
379GLBR-108	22OCT4:203SA20	2/19/92	52.78	50 U	50 U	25 U	25 U	71.2%	106.5%
379GLBR-123	23OCT4:274SA20	2/19/92	99.79	50 U	50 U	25 U	25 U	93.7%	143.2%
379GLBR-136	24OCT10:457SA20	2/19/92	90.15	50 U	50 U	25 U	25 U	58.9%	103.0%
379GLBR-139	24OCT10:508SA20	2/24/92	77.20	60 U	60 U	35 U	35 U	84.5%	133.6%
379GLBR-145	24OCT6:203SA30	2/24/92	56.46	60 U	60 U	120	35 U	98.2%	142.6%
379GLBR-153	25OCT9:214SA30	2/24/92	99.12	60 U	60 U	35 U	35 U	81.9%	141.6%
379GLBR-166	25OCT9:307SA30	2/24/92	91.48	60 U	201	201	35 U	91.5%	125.9%
379GLBR-167	25OCT9:368SA30	2/24/92	99.41	60 U	60 U	67	35 U	87.1%	124.2%
BIN B									
379GLBR-65	09OCT9:542SB10	2/19/92	62.26	50 U	50 U	25 U	25 U	76.5%	149.2%
379GLBR-174	25OCT12:153SB10	2/24/92	62.01	60 U	104	80	35 U	75.6%	125.4%
379GLBR-181	25OCT2:254SB10	2/24/92	99.00	60 U	60 U	94	35 U	78.9%	119.7%
379GLBR-193	25OCT3:527SB10	2/24/92	95.93	60 U	327	262	35 U	90.6%	129.7%
379GLBR-196	25OCT3:578SB10	2/24/92	99.10	60 U	81	75	35 U	105.0%	169.6%
379GLBR-199, Rep 1	30OCT3:573SB20	2/24/92	63.20	60 U	350	371	35 U	73.3%	138.9%
379GLBR-199, Rep 2	30OCT3:573SB20	2/24/92	63.20	60 U	92	96	35 U	68.5%	125.7%
379GLBR-199, Rep 3	30OCT3:573SB20	2/24/92	63.20	60 U	60 U	84	35 U	96.1%	169.1%
379GLBR-210	31OCT9:027SB20	2/24/92	87.81	60 U	319	158	35 U	117.1%	145.8%
379GLBR-213	31OCT9:078SB20	2/24/92	99.79	60 U	60 U	35 U	35 U	112.5%	222.2%
379GLBR-222	31OCT9:244SB20	2/24/92	99.81	60 U	60 U	35 U	35 U	65.4%	167.6%
379GLBR-243	31OCT11:503SB30	2/24/92	62.88	60 U	120	97	35 U	98.6%	164.5%
379GLBR-248	31OCT2:354SB30	2/24/92	99.60	60 U	60 U	35 U	35 U	70.0%	182.4%
379GLBR-268	1NOV10:208SB30	2/24/92	99.60	60 U	60 U	35 U	35 U	66.7%	152.9%
379GLBR-269	1NOV10:157SB30	2/24/92	97.78	60 U	60 U	176	35 U	100.3%	145.0%

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	Concentrations in ng/g dry weight						% Surrogate Recovery	
				Aroclor		Aroclor		Aroclor	Aroclor	Tetrachloro-	Octachloro-
				1242	1248	1254	1260	m-Xylene	naphthalene		
BIN C											
369GLBR-73	09OCT4:562SC10	2/19/92	62.20	50 U	50 U	113	25 U	87.1%	125.3%		
379GLBR-282	18NOV3:503SC10	2/24/92	62.71	60 U	232	35 U	35 U	96.0%	175.7%		
379GLBR-288	18NOV6:1510LC10	2/24/92	73.20	60 U	975	35 U	35 U	104.9%	137.4%		
379GLBR-302	19NOV8:594SC10	3/4/92	99.66	60 U	60 U	17	35 U	103.1%	115.4%		
379GLBR-307	19NOV9:23ISC10	3/4/92	98.79	60 U	60 U	35 U	35 U	71.5%	122.0%		
379GLBR-311	19NOV11:303SC20	3/4/92	69.87	60 U	209	35 U	35 U	97.1%	119.8%		
379GLBR-323, Rep 1	19NOV4:504SC20	3/4/92	99.79	60 U	133	35 U	35 U	97.2%	98.5%		
379GLBR-323, Rep 2	19NOV4:504SC20	3/4/92	99.79	60 U	119	35 U	35 U	101.0%	96.4%		
379GLBR-323, Rep 3	19NOV4:504SC20	3/4/92	99.79	60 U	114	35 U	35 U	95.3%	136.7%		
379GLBR-337	20NOV8:157SC20	3/4/92	95.62	60 U	284	35 U	35 U	102.6%	188.1%		
379GLBR-340	20NOV11:443SC30	3/4/92	55.58	60 U	163	35 U	35 U	67.5%	119.3%		
379GLBR-350	20NOV3:154SC30	3/4/92	95.97	60 U	109	35 U	35 U	72.5%	143.8%		
379GLBR-357	20NOV3:337SC30	3/4/92	77.29	60 U	738	500 U**	35 U	125.7%	90.1%		
379GLBR-360	20NOV3:278SC30	3/4/92	99.58	60 U	60 U	35 U	35 U	75.8%	153.3%		
BIN D											
379GLBR-46	08OCT5:132SD30		60.26	50 U	50 U	25 U	25 U	76.8%	130.3%		
379GLBR-378	21NOV1:353SD10	3/4/92	57.63	60 U	320	35 U	35 U	71.7%	199.9%		
379GLBR-384	21NOV5:073SD10	3/4/92	100.00	60 U	251	35 U	35 U	96.7%	104.6%		
379GLBR-388	21NOV5:107SD10	3/4/92	93.10	60 U	674	35 U	35 U	103.6%	129.0%		
379GLBR-395	21NOV5:224D10	3/4/92	100.00	60 U	60 U	35 U	35 U	86.4%	169.3%		
Blank-1		2/19/92	NA	50 U	50 U	25 U	25 U	46.1%	86.5%		
Blank-2		2/24/92	NA	60 U	60 U	35 U	35 U	77.7%	146.6%		
Blank-3		3/4/92	NA	60 U	60 U	35 U	35 U	86.3%	126.9%		

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	Concentrations in ng/g dry weight				% Surrogate Recovery	
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
STANDARD REFERENCE MATERIAL									
SRM-1 HS-2		2/19/92	NA	50 U	50 U	262	25 U	67.6%	194.2%
SRM-2 HS-2		2/24/92	NA	60 U	60 U	261	35 U	84.0%	114.4%
SRM-3 HS-2		3/4/92	NA	60 U	60 U	381	35 U	83.9%	180.9%
	certified value		NA	NC	NC	111.8	NC	NA	NA
			NA	NC	NC	±2.5	NC	NA	NA
SPIKE RESULTS									
Amount Spiked									
379GLBR-Blank-2+ Spike DUP				NS	NS	2500	NS	NA	NA
Amount Recovered				NS	NS	3173	NS	60.8%	132.9%
Percent Recovery				NS	NS	3173	NS	NA	NA
				NS	NS	127%	NS	NA	NA
Amount Spiked		3/4/92		NS	NS	2500	NS	NA	NA
379GLBR-Blank-3				NS	NS	35 U	NS	86.3%	126.9%
379GLBR-Blank-3 Spike DUP				NS	NS	2767	NS	78.2%	96.2%
Amount Recovered				NS	NS	2767	NS	NA	NA
Percent Recovery				NS	NS	111%	NS	NA	NA
Amount Spiked		3/4/92		NS	NS	2500	NS	NA	NA
379GLBR-Blank-3				NS	NS	35 U	NS	86.3%	126.9%
379GLBR-Blank-3+ Spike DUP				NS	NS	5149	NS	73.8%	243.0%
Amount Recovered				NS	NS	5149	NS	NA	NA
Percent Recovery				NS	NS	206%	NS	NA	NA



**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	Concentrations in ng/g dry weight				% Surrogate Recovery	
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
Amount Spiked									
379GLBR-46	08OCT5:132SD3O	2/19/92	60.26	NS	NS	553	NS	NA	NA
379GLBR-46 + Spike				NS	NS	25 U	NS	76.8%	130.3%
Amount Recovered				NS	NS	455	NS	83.0%	NA
Percent Recovery				NS	NS	455	NS	NA	NA
				NS	NS	82%	NS	NA	NA
Amount Spiked									
379GLBR-46	08OCT5:132SD3O	2/19/92	60.26	NS	NS	714	NS	NA	NA
379GLBR-46 + Spike				NS	NS	25 U	NS	76.8%	130.3%
Amount Recovered				NS	NS	517	NS	85.0%	126.8%
Percent Recovery				NS	NS	517	NS	NA	NA
				NS	NS	72%	NS	NA	NA
Amount Spiked									
379GLBR-268	1NOV10:208SB3O	2/24/92	99.60	NS	NS	547	NS	NA	NA
379GLBR-268 + Spike				NS	NS	62	NS	66.7%	152.9%
Amount Recovered				NS	NS	732	NS	63.6%	137.6%
Percent Recovery				NS	NS	670	NS	NA	NA
				NS	NS	122%	NS	NA	NA
Amount Spiked									
379GLBR-268	1NOV10:208SB3O	2/24/92	99.60	NS	NS	480	NS	NA	NA
379GLBR-268 + Spike				NS	NS	62	NS	66.7%	152.9%
Amount Recovered				NS	NS	696	NS	75.7%	138.2%
Percent Recovery				NS	NS	634	NS	NA	NA
				NS	NS	132%	NS	NA	NA

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	Concentrations in ng/g dry weight				% Surrogate Recovery	
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
Amount Spiked									
379GLBR-388	21NOV5:107SD10	3/4/92	93.10	NS	NS	534	NS	NA	NA
379GLBR-388 + Spike				NS	NS	674	NS	103.6%	129.0% *
Amount Recovered				NS	NS	1157	NS	115.8%	106.4%
Percent Recovery				NS	NS	483	NS	NA	NA
				NS	NS	90%	NS	NA	NA
Amount Spiked									
379GLBR-388	21NOV5:107SD10	3/4/92	93.10	NS	NS	585	NS	NA	NA
379GLBR-388 + Spike				NS	NS	674	NS	103.6%	129.0% *
Amount Recovered				NS	NS	1141	NS	115.0%	79.0%
Percent Recovery				NS	NS	467	NS	NA	NA
				NS	NS	80%	NS	NA	NA
<b>REPLICATE ANALYSIS</b>									
379GLBR-199, Rep 1	30OCT3:573SB20	2/24/92	63.20	60 U	350	371	35 U	73.3%	138.9% *
379GLBR-199, Rep 2	30OCT3:573SB20	2/24/92	63.20	60 U	92	96	35 U	68.5%	125.7% *
379GLBR-199, Rep 3	30OCT3:573SB20	2/24/92	63.20	60 U	60 U	84	35 U	96.1%	169.1% *
	RSD %			NA	117% *	89% *	NA	NA	NA
379GLBR-323, Rep 1	19NOV4:504SC20	3/4/92	99.79	60 U	133	63	35 U	97.2%	98.5%
379GLBR-323, Rep 2	19NOV4:504SC20	3/4/92	99.79	60 U	119	51	35 U	101.0%	96.4%
379GLBR-323, Rep 3	19NOV4:504SC20	3/4/92	99.79	60 U	114	51	35 U	95.3%	136.7% *
	RSD %			NA	8%	13%	NA	NA	NA

U = Not detected at detection limit shown.

J = Detected below detection limit.

D = Detected Aroclor, but undetected pattern due to low levels.

\* = Outside of QC limits.

\*\* = Matrix interference.

NS = Not Spiked. NC = Not certified.

NA = Not Applicable.

RSD % = Relative Standard Deviation.

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES  
TABLE NO. 2**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	(concentrations in ng/g dry weight)				% Surrogate Recovery	
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
379GLBR-15, Rep 1	07OCT1:212SCO	10/10/91	59.0	73 E	217	99 E	20 U	53.9%	85.1%
379GLBR-15, Rep 2	07OCT1:212SCO	10/10/91	59.0	79 E	236	108 E	20 U	56.8%	89.2%
379GLBR-15, Rep 3	07OCT1:212SCO	10/10/91	59.0	73 E	239	20 U	20 U	56.9%	95.9%
379GLBR-17	07OCT12:091SAO	10/10/91	57.9	20 U	85	20 U	20 U	60.9%	93.2%
379GLBR-21	07OCT12:531SBO	10/10/91	55.2	20 U	20 U	20 U	20 U	66.9%	107.3%
379GLBR-22	07OCT1:521SDO	10/10/91	58.7	20 U	179 E	202	20 U	66.7%	100.6%
Blank		10/10/91	NA	20 U	20 U	20 U	20 U	82.9%	127.4% *

**STANDARD REFERENCE MATERIAL**

SRM-1 (1941)	certified value	10/10/91	NA	20 U NC	395 NC	315 NC	153 NC	77.9% NA	123.1% * NA
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**SPIKE RESULTS**

Amount Spiked				NS	NS	880	NS	NA	NA
379GLBR-22	07OCT1:521SDO	10/10/91	58.7	NS	NS	202	NS	66.7%	100.6%
E79GLBR-22 + Spike				NS	NS	689	NS	76.7%	121.3% *
Amount Recovered				NS	NS	487	NS	NA	NA
Percent Recovered				NS	NS	55%	NS	NA	NA

**BUFFALO RIVER PILOT PROJECT (CF #379)  
PCB/AROCLORS IN SEDIMENT SAMPLES  
TABLE NO. 2**

MSL Code	Sponsor ID	Extraction Date	Dry Wt. (%)	(concentrations in ng/g dry weight)				% Surrogate Recovery	
				Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
REPLICATE ANALYSIS									
379GLBR-15, Rep 1	07OCT1:212SCO	10/10/91	59.0	73 E	217	99 E	20 U	53.9%	85.1%
379GLBR-15, Rep 2	07OCT1:212SCO	10/10/91	59.0	79 E	236	108 E	20 U	56.8%	89.2%
379GLBR-15, Rep 3	07OCT1:212SCO	10/10/91	59.0	73 E	239	20 U	20 U	56.9%	95.9%
		RSD %		5%	5%	9%	NA	NA	NA

U = Not detected at detection limit shown.  
 E = Estimated/most likely due to residual peaks of primary aroclor.  
 \* = Outside of laboratory control limits of 40-120%.  
 NA = Not Applicable.  
 NS = Not Spiked. NC = Not certified.  
 RSD % = Relative Standard Deviation.